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Conquer Crystallization Challenges

Question the need for cooling and ensure you have the right data

By Tom Blackwood, Contributing Editor

Crystallization is easy! You just cool the solution and out drops your product. That's a delusion as numerous plants can attest. Many products that start out in this manner cause problems in downstream processing. For instance, fine crystals may not separate from the solvent completely and drying may take a long time.

Once the chemists have given you a process — react, cool, separate and dry — they should look at various scenarios and alternative routes to the product. Such alternatives certainly exist. Cooling may not be the optimum way to generate supersaturation.

So, let's examine five alternative routes that you should suggest to the chemists:

1. *Generate enough supersaturation to nucleate the product and slowly grow the crystals to a large size.* Sometimes this means an incubation period or even a fines-destruction step to ensure the correct number of nuclei. The crystals should be big enough to separate from the solvent easily and even to allow their washing. With less solvent to evaporate, drying can be more rapid. Often, the equipment required for the downstream processing is less expensive and smaller. Generally, this is a big benefit. However, spending a lot of time in the crystallization process to make a large particle can destroy that advantage.

2. *Generate the supersaturation and introduce seeds or artificial nucleation via sonication to control the number of nuclei.* This extra step may make the solvent easier to remove, even with a smaller crystal. This results in less time spent crystallizing while still realizing a reduced drying time.

3. *Remove the excess nuclei generated by primary or secondary nucleation via fines destruction, and return that solute to the*

crystallizer. This will allow operating at a higher supersaturation that will increase growth rate, reduce crystallization time, and maintain the advantages cited in the first route. An optional way to remove excess fines is through Ostwald ripening, where the batch is held at the end of crystallization to re-dissolve fine particles onto larger crystals.

4. *Rather than making a large crystal, stick with the fine product and perform multiple crystallizations using the solvent in a cascade manner to wash the crystals or even use liquid/liquid extraction to purify the wash.* The fine particles produced can be agglomerated during drying or pelletized through extrusion to give granular product of the desired size.

5. *Remove solvent instead of cooling.* While cooling works well for many chemicals, some have unusual, if not strange, solubility curves. In such cases, you must remove solvent to generate supersaturation. Also, polymorphs can complicate the crystallization, especially if the solubility curves cross at some temperature. Solvent evaporation often makes more sense than cooling when considering options such as fines destruction. Addition of anti-solvents also may outperform other means of generating supersaturation.

You should evaluate, at least on paper, all these routes before selecting a process design or type of crystallizer.

Many process designs run into problems when the meta-stable-zone width isn't well defined or understood. Indeed, I've seen numerous difficulties occur because of this oversight. You should generate solubility curves both from solution and dissolution of the product to determine this no-man's land of solubility. Such curves will help any manufacturer of crystallizers make the best suggestion for a device. Also, this information will ease grappling with future process or quality problems.

The most overlooked physical properties in a crystallization process are growth and nucleation rate. Growth often is expressed as time to reach an average size and never has been studied as a function of supersaturation and particle size. I realize that both nucleation and growth aren't easy to understand, but an analysis of the particle size distribution gives a starting point to estimate growth and evaluate other routes that might provide a better and cheaper product.

The alternatives suggested above don't address one of the most critical choices made in the process design: whether to opt for batch or continuous crystallization. Plants often make this choice based on upstream or downstream operations along with what equipment is available or has been used in the past. However, sometimes changing upstream/downstream operations can lead to an overall process that provides optimum quality and cost. ■

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Beware of Blending Myths

But first determine if blending really is required

By Tom Blackwood, Contributing Editor

The problem with particulate solids is they segregate every time we turn around. Why? Because they are a two-phase material and only about half is the solid we're interested in. In my days as a troubleshooter at corporate engineering, the most common call I got from plants was about blenders that didn't blend. The problems arose because people believed some myths.

The most common myth is that increased blending time results in a better blend. One of our customers mixed an inert ingredient with our product in a ribbon blender. Prior production with a different active ingredient gave an acceptable blend in 15 minutes — so the customer used that time for the new product. The resulting mix was highly variable in composition. To compensate, the plant increased the blend time to a half hour and then an hour with no improvement. In fact, the mix got worse. We found that seven minutes gave a perfect blend. What happened was that friction with the inert ingredient caused a surface charge to develop on the new active ingredient; this friction was unexpected. Most materials reach a perfect blend in a very short period of time.

Another common myth is that all blenders are created equal. We made a catalyst by an extrusion process that gave a slightly variable particle size. The catalyst was to be put

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into very long tubes that had to have the same pressure drop; blending was believed to smooth the distribution so the pressure drop would be uniform. Without conducting any flowability tests, the plant opted for an available twin-cone blender with 45° walls. Even after changing blend time, the product came out in linear order of size. Testing for angle-of-slide showed the 45° wall held the smaller particles and concentrated them on the top of the mix. Another twin-cone blender with a steeper cone and an internal ribbon solved the problem.

The idea that fluidization will mix solids well is another myth. Density and particle size determine how easily a material will fluidize or defluidize — a "Geldart" classification often is used as an indicator. Several ingredients were added to a blender that operated at very high speed to mix the materials. The speed was dropped to a crawl to aid in discharge. The mixture deaerated slowly and the ingredients separated almost in layers. All the ingredients but one were Geldart Group A. To demonstrate how the mixture responded, I put it in a graduated cylinder, which I shook. Then, I dropped into the mix a coin, which went all the way to the bottom. A half-hour later, I dropped another coin, which went half way down. It took two hours before I could drop a coin and have it stay on top. Jogging the blender (short fluidization times) and increasing the discharge speed maintained the blend and avoided segregation. In this case, fluidization was working well but keeping the solids fluidized was the real problem.

Many other myths exist involving agitator type, multiple ribbons and attrition in blenders. It's hard to mechanically move particulate solids without incurring some sort of damage, so it's often better to avoid blending. However, sometimes attrition in a blender can be put to good use. We had developed a new disinfectant product that outperformed our current offering. However, it was lighter and caused problems for the formulator. We added a twin-ribbon blender to the production line to slightly grind the product, which allowed the final material to match the density of the former product.

Not only dry blenders suffer from these types of problems. Crystallizers, solid/liquid mixers and conveyors can have similar issues. Particulate solids want to settle in a fluid whether gas or liquid, so always keep that in mind. ■

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Solve the Mystery of Time Effects

Conduct a time consolidation test to determine the flow performance of a bulk solid

By Vinnie Hebert, Ametek Brookfield

On the surface, the flow of powders through gravity feed systems seems easy and straightforward. Fill a vessel with a bulk solid and open the chute. The powder flows out and profits flow in. In a perfect world, this would happen every time. The reality is, due to the complexity of bulk solids, flow problems such as jams, erratic discharge, segregation and rat-holing can occur. To understand how to handle and process powders, you need to identify their flow properties.

Characterizing powders is most commonly done when they are initially defined in R&D, during a pilot production run or in a more complex process involving blending. What is often overlooked are time effects on the material and how this affects its flow properties. That is, how long has the material sat in a warehouse? How long has it taken for the material to be transported? How will the self-compaction of the powder affect its flow properties? To properly address time effects on powders, a relevant test needs to be performed which simulates what happens to a powder over time.

SHEAR CELL TESTING

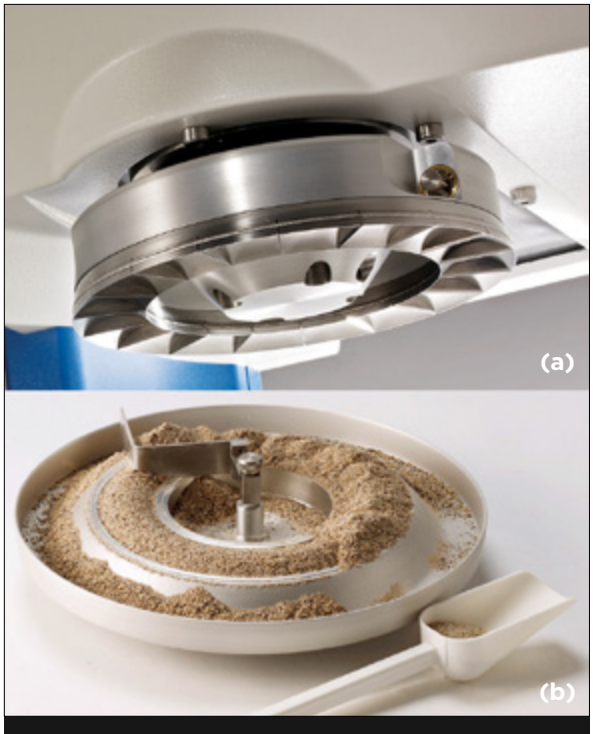
For characterization of powder flow properties pertaining to bulk solids in gravity flow, shear cell testing is the accepted scientific method. An annular shear cell can acquire flow function data quickly and accurately by shearing material at defined consolidation stresses and measuring the inter-particle strength. By plotting this data via Mohr circle analysis, accurate, repeatable flow

data can be acquired. This data includes flow function, bulk density (both loose fill and final compaction state), and internal friction angle. Calculations for potential arching dimension and rat-hole diameter can be derived from this data. Analysis of this data is used to predict the general flow properties of the bulk solid in gravity discharge from a hopper (See Figure 1).

Characterizing the flow of a bulk solid in R&D or during pilot production runs gives data on how the material will flow when it is freshly made and has not undergone the consequence of significant settling in the containment vessel. A time consolidation test also utilizes the same shear cell and, as its name implies, tests the flow properties of the same bulk solid over a specific period of time (Figure 2). Let's investigate why this is important.

TIME EFFECT ON BULK SOLIDS

It may not seem obvious, but the longer a material sits in storage or undergoes transport, its flow properties may have the potential to change. For some materials, sitting undisturbed for a long period of time



SHEAR CELL TEST

Figure 1. An annular shear cell tester (a) can acquire flow function data quickly and accurately by shearing material (b) at defined consolidation stresses and measuring the inter-particle strength.

POWDER FLOW TESTER

Figure 2. This powder flow tester uses an annular shear cell to measure flow properties of bulk solids.

will not affect flow properties; for others, a few hours can be the difference between flowing and jamming.

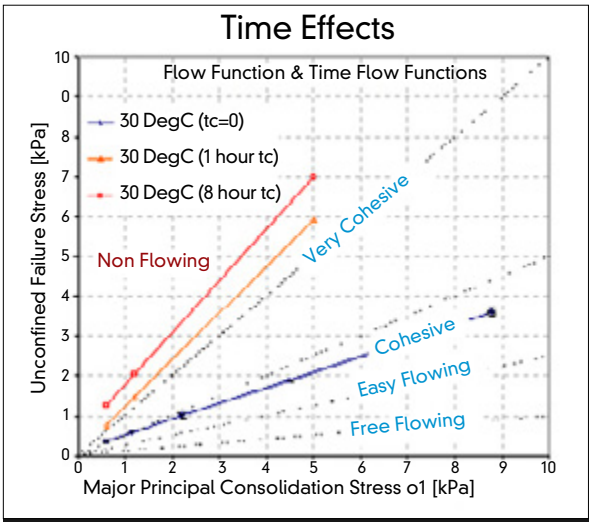
For example, assume that a material has been characterized for common flow properties with an instantaneous flow function test. Information on flow function, arching dimension, bulk density has been acquired by testing with the annular shear cell and used to create an operating procedure for powder processing. Production begins and the material flows easily. During the course of production, a maintenance issue arises and a shutdown in production occurs until this issue is resolved. This takes a few hours. Once completed, production is restarted, but the material will not flow. Efforts to loosen the material are not working; the material has consolidated and does not want to flow. Further downtime takes place while the material is removed from the feeder system, broken up, reconstituted and then production restarted.

Certainly, it would have been easier to identify this issue beforehand and takes steps to resolve it. This is where a time consolidation test utilizing an annular shear cell comes into play.

TIME CONSOLIDATION TESTING

As previously stated, a time consolidation test will evaluate bulk solids flow behavior over a period of time. To implement time consolidation analysis of a material, a common way to begin characterization is to run the instantaneous flow function followed by a repeat of the test 12 hours later. If it is discovered that the material becomes non-flowing after 12 hours, the test is then scaled back to discover the break point of the material; that is, how long did it take for the material to consolidate and become non-flowing. The time consolidation test is then done at shorter time intervals — 10 hours, 8 hours, 6 hours, etc. — to determine the break point. Once the break point is determined, an operating procedure for material handling in case of work stoppages can be written accordingly.

Consider the time consolidated flow function graph in Figure 3. This material is



TIME CONSOLIDATION RESULTS

Figure 3. Results show this material is cohesive in nature for instantaneous flow. If this material sits in the feeder system for one hour, it will become non-flowing and jam the system.

cohesive in nature for instantaneous flow. If this material is allowed to sit in the feeder system for 1 hour, it will become non-flowing and jam the system. Once production is halted, steps need to be immediately taken to address the self-compacting nature of this product. In a case like this, the solution might be to simply remove the material from the system or insert a mixing device that will keep the material moving until the flow process can be restarted.

Another example might be a test that reveals a powder becoming non-flowing in 6 hours. The standard operating procedure might then be conservatively written to define that the material should not sit in the feeder system for more than 4 hours. This will ensure proper flow in up to 4 hours when the system is restarted. If longer than 4 hours, then the SOP will define the necessary steps (removal, a mixer, vibration) for the product.

CONCLUSION

This time consolidation test, while often overlooked, may be one of the most important tests that can be run on a bulk solid. By identifying a material's flow properties over a time period when movement of powder does not take place, expensive downtime due to jamming can be minimized or eliminated. Furthermore, this test can be used to identify what happens to materials in transit or storage; the fact that powders continue to consolidate when left in a static condition is the phenomenon that is important to understand. Use of time consolidation testing is the best approach to quantifying the challenge that a powder will present when initial discharge takes place after a time period of no movement.

VINNIE HEBERT is sales manager – Powder Flow Tester for Ametek Brookfield, Instrumentation & Specialty Controls Division. He can be reached at vinnie.hebert@ametek.com.

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Handle Water Vapor within Oil-Sealed Vacuum Pumps

Evaluate several methods for condensing water vapor

By Phil Vibert, Tuthill Vacuum & Blower Systems

Water vapor is not a permanent gas so it can condense within the pump during compression. If this occurs, it can contaminate the oil leading to reduced net capacity due to higher vapor pressure. This can also increase pump wear or cause pump seizure due to the reduced oil lubricity. Also, the same mass flow of water occupies 60% greater volume in the vapor phase than at the same pressure and temperature because V1 / V2 ≈ MW2 / MW1 and for water and air, V1 ≈ 29 / 18 V2 = 1.61 V2 where V1 = volume of water vapor and V2 = volume of air.

OPTIONS FOR HANDLING WATER VAPOR

There are two main options for handling water vapor within oil-sealed vacuum pumps. These options include preventing the water vapor from condensing within the pump, or to condense the water vapor out ahead of the pump. Each option offers different methods of carrying out the process.

Elevated oil temperature, gas ballasting, air stripping or a vapor handling system can help prevent water vapor from condensing within the pump. Using a pre-condenser or a cold trap condenses water vapor out ahead of the pump. Table 1 compares each method and more details on each are below.

ELEVATED OIL TEMPERATURE

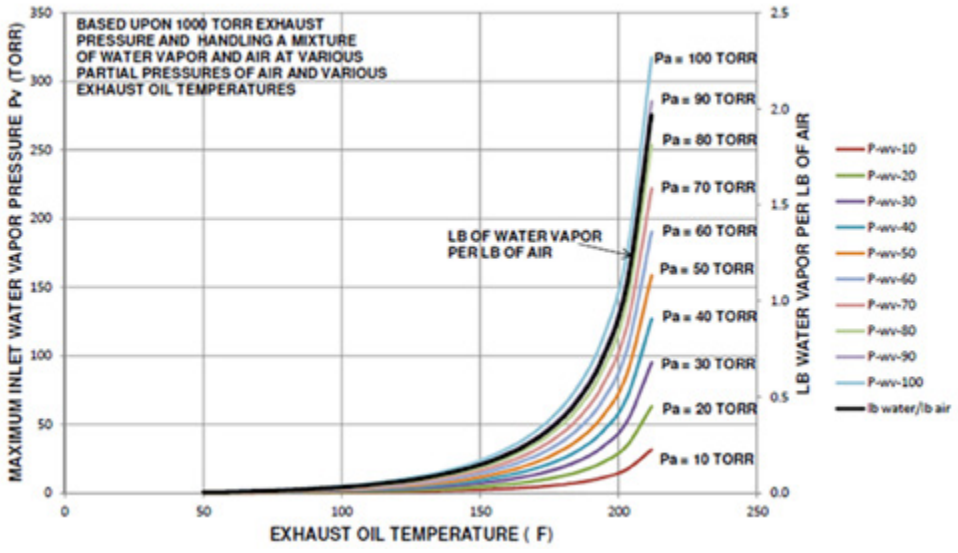
Process gas flow consists of a mixture of permanent gas, such as air plus water vapor, entering the pump. To avoid condensing within the pump:

$$(Pv / P\ total)\ @\ inlet < (Pv\text{-}sat / Pd)\ @\ exhaust$$

where $P\ total = Pv + Pa$ and $Pd = P_{Atmos} + P_{Discharge\ Valves} + P_{OME}$.

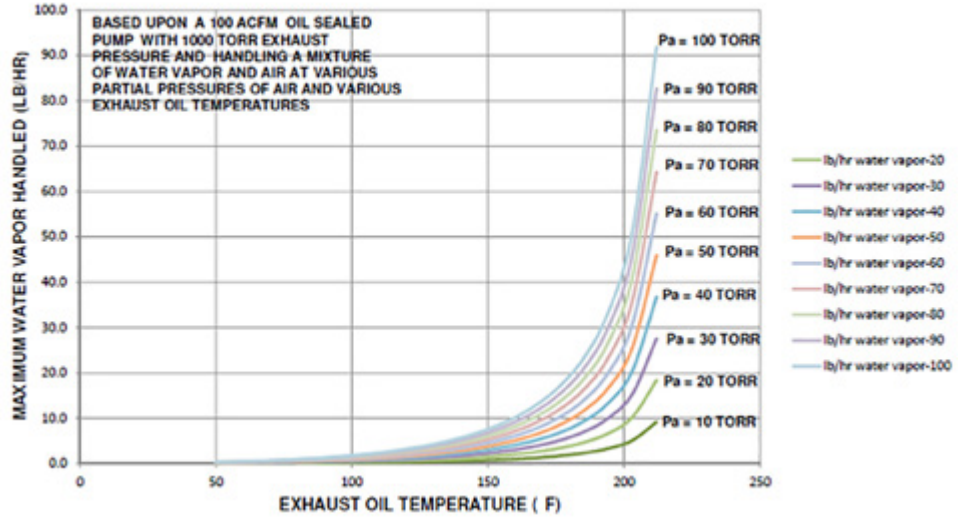
Regulating the exhaust oil temperature may be sufficient to prevent condensing within the pump and to maintain the water in the vapor phase as it passes out of the exhaust (See Figures 1 and 2).

Using an OME (oil mist eliminator) might require collecting coalesced oil in a separate container. This would be the method as opposed to using a suckback line to pump



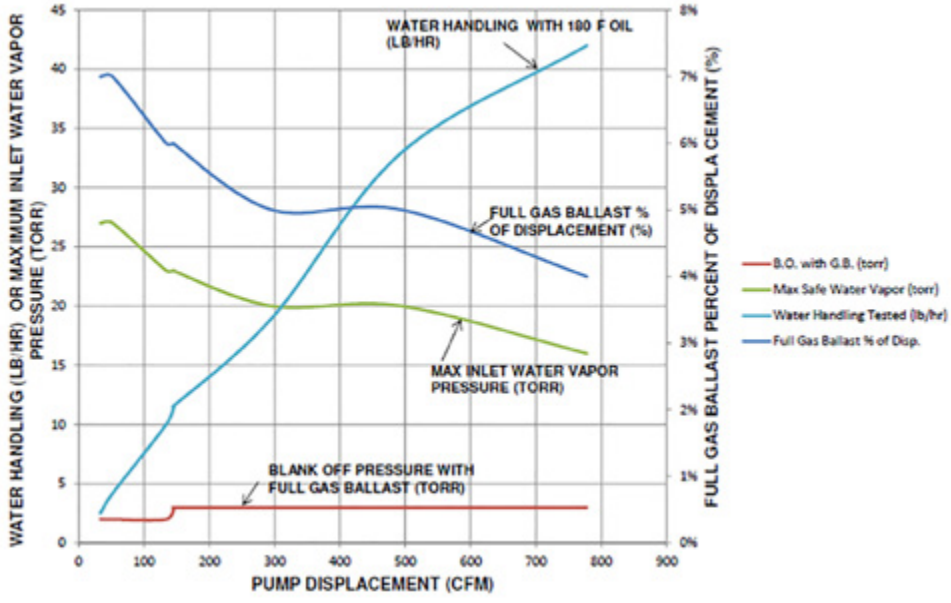
MAX INLET WATER VAPOR PRESSURE

Figure 1. The maximum inlet water vapor pressure for oil sealed pumps is identified in this chart. This level is measured as the pumps handle a mixture of water vapor and air using various partial pressures of air and oil temperature.



WATER VAPOR CHART

Figure 2. This chart identifies the maximum pounds per hour of water vapor handled without condensing by 100 CFM oil sealed pumps. This happens while handling a mixture of water vapor and air at various inlet partial pressures of air and oil temperatures.



ROTARY PISTON PUMP PERFORMANCE

Figure 3. This chart depicts the performance of single-stage oil-sealed rotary piston pumps handling water vapor using a full gas ballast.

suction. A drop-out leg would be used at the OME exhaust piping because the OME housing or exhaust piping could act as a condensing surface if it is not heated, or if the temperature drops sufficiently.

The desirable oil properties for this method include good demulsibility (separation), rust and oxidation additives, resistance to thermal degradation, and high viscosity index (VI) if used in rotary piston or vane vacuum pumps for lubrication to reduce changes in viscosity with temperature.

GAS BALLASTING

A gas ballast is normally standard on all rotary piston and vane vacuum pumps (Figure 3). This method admits air into the compression stroke of the pump. The entering air is heated while passing through the pump and can hold more water vapor before becoming a saturated mixture. A gas ballast can be used to handle 100% of the water vapor entering the pump, up to its water vapor tolerance (Figure 4).

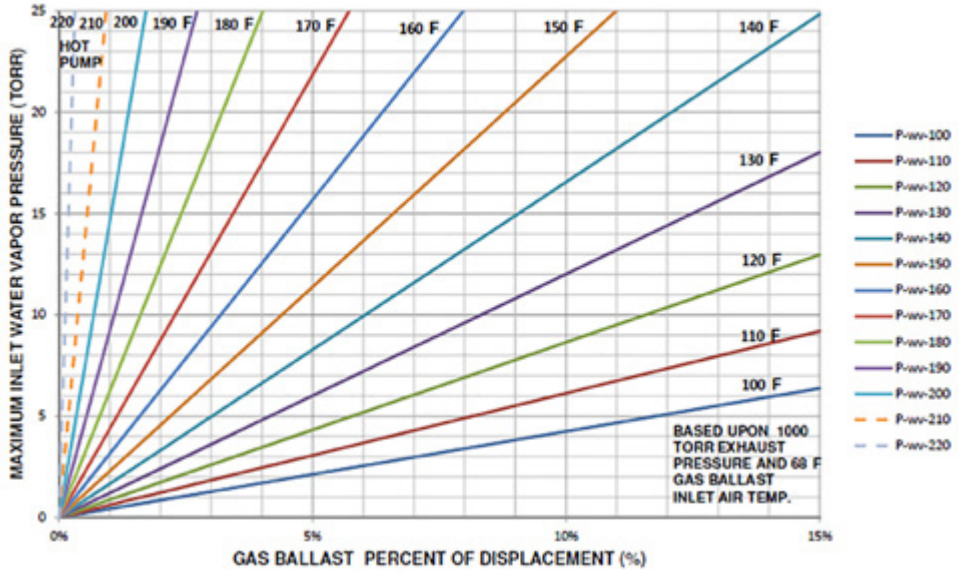
The quantity of gas ballast required (Figure 5) to handle a given amount of water vapor pressure Pv without condensing in the pump is determined from:

$$P1\ V1/T1 = P2\ V2/T2 \text{ or}$$

$$P\text{-}gb\ V\text{-}gb / T\text{-}gb = Pd\ Vd / Td$$

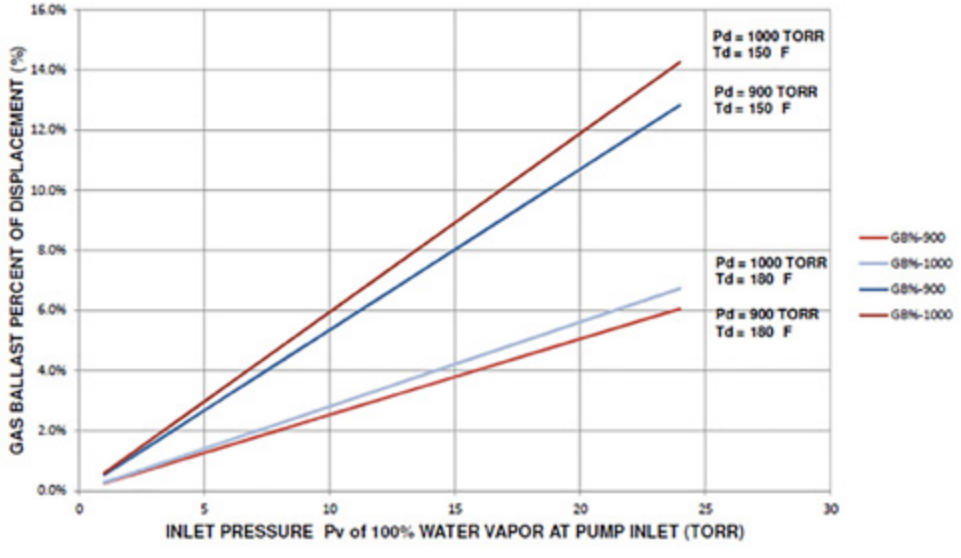
and $(Pv / P\ total)$ @ inlet <

$(Pv\text{-}sat / Pd)$ @ exhaust



GAS BALLAST WATER VAPOR PRESSURE LEVELS

Figure 4. The maximum amount of inlet water vapor pressure for oil sealed pumps is charted here. These levels are reached while the pumps handle 100% water vapor using various gas ballast percentage of displacement and exhaust pump temperatures.



GAS BALLAST REQUIRED

Figure 5. The gas ballast percentage required for handling various inlet pressures of 100% water vapor using single-stage oil-sealed rotary piston pumps is charted above.

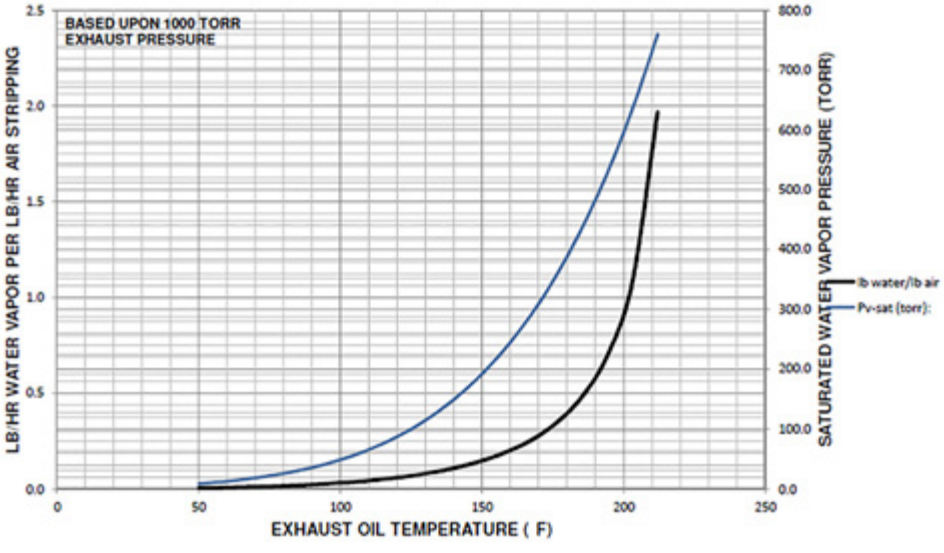
If 20 Torr water vapor is handled at suction with 180°F exhaust oil temperature, then Pv-sat @ 180°F is 388 Torr and the water vapor volume can't be compressed > Pv-sat-disch / Pv-inlet > 388/20 > 19/1 to prevent condensing during discharge.

Vd = P-gb V-gb Td /(Pd T-gb) = (760)(V-gb) (640°R) / (900)(530°R) = Vd = 1.02V-gb and if V-gb = fD then Vd = 1.02fD but if the water vapor volume at discharge Vd can't be compressed more than 19/1 = Pd/P1 and Vd = P1D/Pd where S1≈D then Vd ≈ 1/19D ≈ 0.92fD and f ≈1/[(19)(1.02)] ≈ 0.05 where f ≈ 0.05 is the faction of gas ballast required or 5% of the pump displacement D based upon Pd = 900 Torr, Td = 180°F, gas ballast P-gb = 760 Torr (Atmos) and T-gb = 70°F.

AIR STRIPPING

The simple addition of air flow to a vacuum pump can remove water vapor. Air can be admitted at suction, interstage or discharge. "Knox air stripping" is the name given to the process of blowing dry compressed air over the discharge valves of rotary piston vacuum pumps in the valve deck cavity.

This process is less efficient than gas ballasting, but a greater air flow can be used while not increasing the inlet pressure (Figure 6). Air stripping can be used on a two-stage, oil-sealed liquid ring vacuum pump by admitting atmospheric air through the attenuation valve which enters the interstage, or through the pump suction like an anti-cavitation bleed. The addition of an air ejector stage to the oil-



AIR STRIPPING CHART

Figure 6. This chart illustrates pounds per hour of water vapor that is handled per pounds per hour air stripping at various discharge temperatures.

sealed liquid ring inlet can provide air stripping as well as provide a lower pressure.

VAPOR HANDLING SYSTEM

Water or other vapors enter a rotary piston pump as a gas. The vapor stays in the gaseous state and any droplets of liquid actually evaporate within the separator housing. The liquid finally condenses within the liquid ring vacuum pump which is full of compatible liquid. A heat exchanger keeps sealant cool and removes heat from condensate. Shell and tube construction prevents contamination of the cooling liquid. The principle is similar to vacuum distillation.

Maximum inlet pressure for Std VHS is normally ≤ 25 Torr.

Water vapor can be handled from P ≤ 25 Torr or higher depending upon the selection of a liquid ring vacuum pump.

PRE-CONDENSER OR COLD TRAP

A pre-condenser with condensate tank is placed ahead of an oil sealed pump. The coolant temperature (Tc) for the pre-condenser must be sufficiently below inlet water vapor temperature (T-in) to allow condensing of the water vapor portion without freezing:

$$34°F < Tc < T\text{-in where } Pv\text{-sat } @ Tc+10 << Pv @ T\text{-in}$$

An optional bypass piping for batch operation can avoid re-evaporation of condensate

	Elevated Oil Temperature	Gas Ballasting	Air Stripping	Vapor Handling System	Pre-Condenser	Cold Trap
Pressure Range	Limited by pump operating pressure range	<20-27 Torr for 100% water vapor Limited by pump operating pressure range for air and water mixtures	Limited by pump operating pressure range	<20-50 Torr	Limited by pump operating pressure range	<0.1 Torr
Does it Change Inlet Pressure	No	Yes	No, if added at discharge (Knox Air Stripping) Yes, if added at inlet	No	No	Yes
Advantages	No additional gas is added. Discharge temperature is increased by regulating cooling water flow.	Adds air to the compression stroke of the rotary piston or vane pump so that the inlet pressure is less affected, compared to addition at inlet. It is more efficient than air stripping.	Addition of air stripping at discharge does not change inlet pressure and mass flow can be larger.	Can be used for inlet pressure <25 Torr without affecting inlet operation.	Larger surface areas and cooling flows can handle larger mass flows of vapor.	More effective for low pressures and small mass flows.

COMPARISON OF METHODS

Table 1. Compare the six methods to help determine which application works best.

at lower pressures. Monitoring of inlet pressure and temperature to pre-condenser is recommended.

Coolant temperature (Tc) for a pre-condenser must be sufficiently below inlet water vapor temperature, T-in, to allow condensing of the water vapor portion without freezing:

$$34°F < Tc < T\text{-in where Pv-sat @ } Tc+10 << Pv \text{ @ } T\text{-in}$$

The condensate tank must be used below the pre-condenser to collect condensed water with a level switch or sight gauge and isolation, and drain valves for periodic draining. For batch processes, the pressure must be monitored. The pre-condenser or condensate tank is isolated from the vacuum pump and a bypass pipe around the condensing portion is used as the pressure drops, as the process water load is removed, to avoid re-evaporation of the condensed water.

Cold traps using refrigerated coolant, or LN_2, can only be used at lower pressures, < 0.1 Torr because cold traps rely on freezing the water vapor to the cold trap surface. They are limited by the heat transfer surface area, flow regime and thermal transfer, and conductance.

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

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NEW!! Minute Clinic

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

Ask the Experts

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

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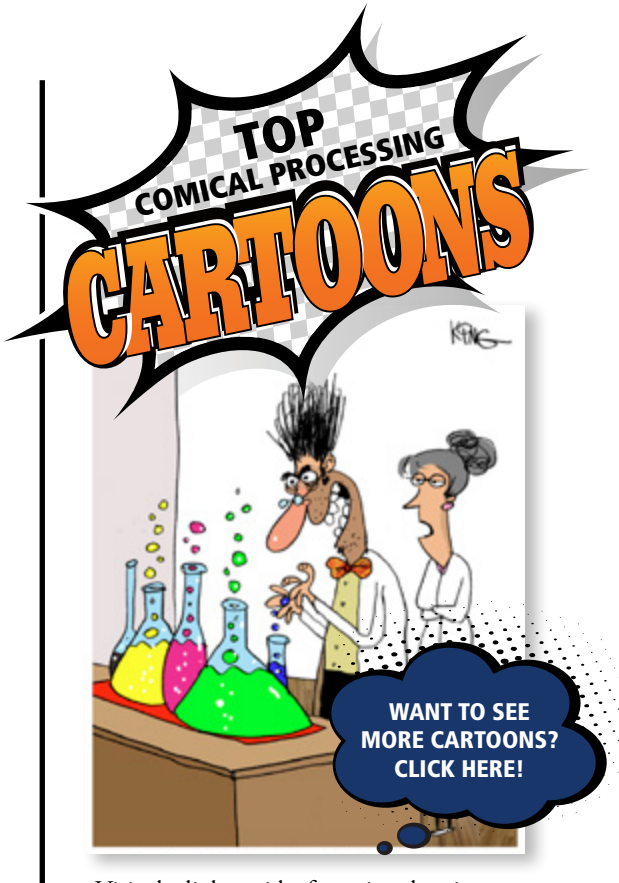

Visit the lighter side, featuring drawings by award-winning cartoonist Jerry King. Click on an image and you will arrive at a page with the winning caption and all submissions for that particular cartoon.