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April
2017

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Fertilizers

Safety Sensors

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Perspectives on Drying

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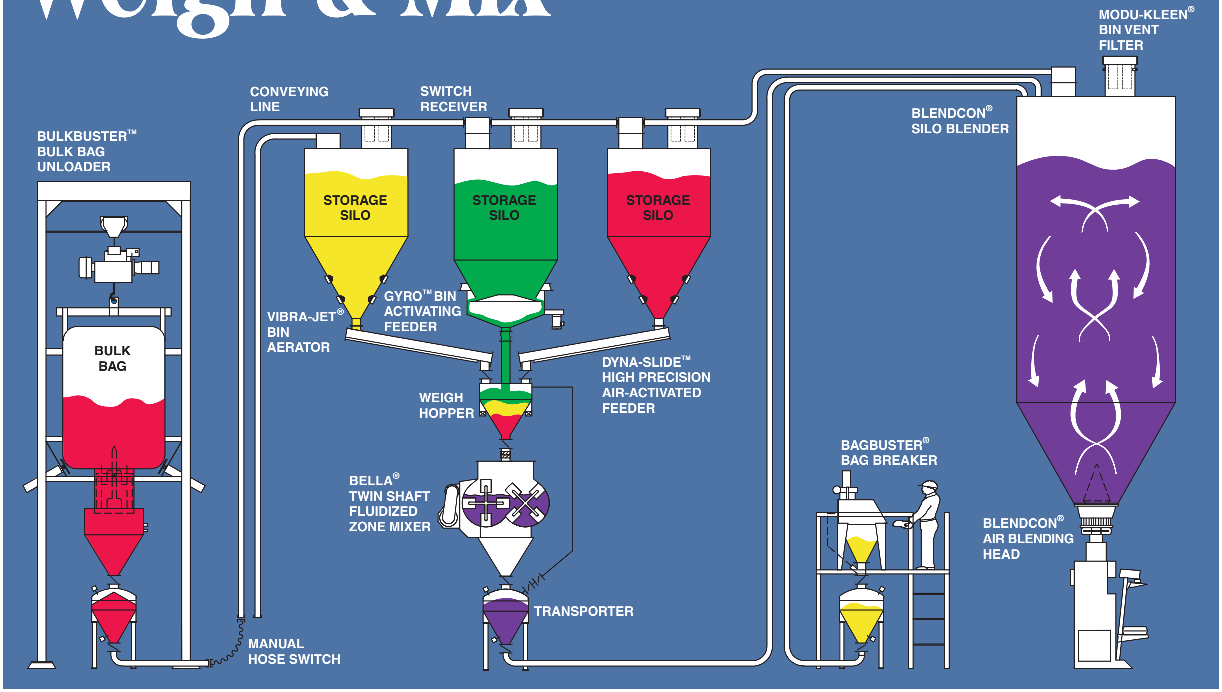
Access Intelligence

The Smart Way to Unload, Convey, Weigh & Mix



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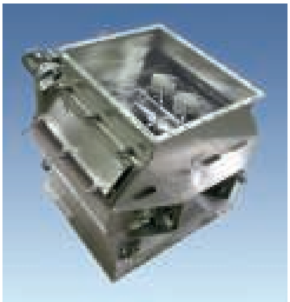

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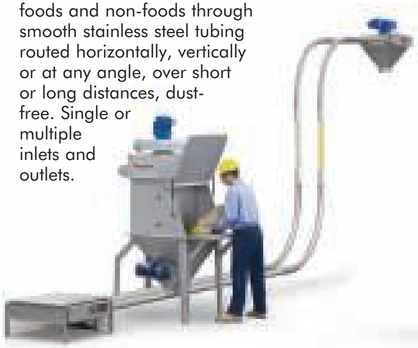

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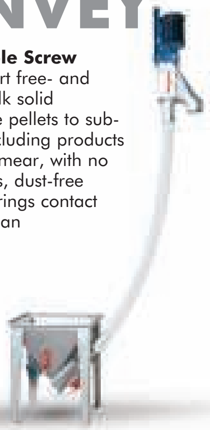

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Editor's Page

Designating a day for water

Last month, special recognition to water-related issues was given on March 22, the date designated by the United Nations as "World Water Day." The first World Water Day was observed in 1993, and has been celebrated every year since. The purpose of the observance is to bring attention to our water resources and offer learning opportunities, as well as to inspire others to sustainably manage those resources. This year, the theme of World Water Day was wastewater, to focus on the need to improve water reuse.

We cover water treatment regularly in our magazine, and I have written about its importance a number of times, yet each time I learn more, it impresses upon me how far-reaching the impact of water-related issues is. The World Water Day website (www.worldwaterday.org) offers the following information in its "Fact Sheet."

- Over 80% of wastewater generated by society goes back into the ecosystem without being used or recycled. High-income countries treat around 70% of wastewater, and the percentage drops dramatically in lower-income countries
- 1.8 billion people are at risk for life-threatening diseases because of contaminated sources of drinking water
- The portion of the world's population living in cities is expected to grow from 50% to 70% by 2050, while many cities are not equipped to address growing wastewater management needs

The CPI contributes

Industrial production accounts for nearly 25% of global water consumption, according to a statement released in conjunction with World Water Day by Lanxess (www.lanxess.com). The chemical process industries (CPI) are well equipped to provide solutions to industrial, as well as municipal water- and wastewater-treatment challenges. Our engineers and scientists are very familiar with pertinent technologies, such as advanced filtration techniques (see the Feature Report in this issue on Crossflow Membrane Filtration, pp. 49–59). And new technologies are being developed around the world. See for example, the Chementator article in this issue about a group in Singapore who are working on removing pollutants from water with iron hydroxides (p. 12), and the work in Korea on developing a new water-treatment adsorbent reported last month (A New Adsorbent for Wastewater Treatment, *Chem. Eng*. March 2017, p. 11).

Spotlighting World Water Day

CPI companies have joined in the observance of World Water Day to help spread the news about water scarcity issues and how to address them. The Dow Chemical Company (www.dow.com) for example, participated in an event in Tucson, Ariz. where representatives from industry, business, academia and government interacted on water-related topics. The event included a tour of the University of Arizona's Water and Energy Sustainable Technology Center, as well as the unveiling of the World Water Day paint scheme for the No. 3 Dow Chevrolet car, which competed in the Camping World 500 at the Phoenix International Raceway on March 19.

Current world news has many concerned about the future of environmental endeavors. The need to share information about the importance of better wastewater treatment is perhaps greater that it has been, making a designated day all the more important. ■

Dorothy Lozowski, Editorial Director

Letters

Steam Concepts

I enjoy reading your magazine and find many of its articles informative and helpful. I noticed several errors in the [Facts at Your Fingertips] article, Steam Concepts, [February 2017, p. 32] and wanted to bring them to your attention:

1. In the section "Pressure-temperature relation" the author states: "enthalpy, a thermodynamic property of a fluid that is defined as the internal energy of the fluid added to the product of its pressure and temperature." This is incorrect. Enthalpy is the internal energy added to the product of its pressure and specific volume.
2. In the second paragraph under "Industrial practice" the author states: "Lower heat transfer rates occur because condensed water can form a film on heat-transfer surfaces, which reduces heat transfer because thermal conductivity of water is much lower than that of steam." The observation is correct but the reasoning is incorrect. The thermal conductivity of liquid water is in fact significantly higher than steam. This is true for most liquids when compared to gases. The reason for the lower heat transfer rates when a film of liquid water forms on the heat transfer surfaces is as the author stated earlier in the article: the heat transfer coefficient (capacity) for condensing steam is significantly higher than for liquid water (at the same temperature and pressure). And this higher heat-transfer capacity is mainly due to it changing phase from a gas to a liquid where large amounts of energy are released. So in fact liquid water must form when steam is condensing on heat transfer surfaces. The trick is to design the surfaces so that this condensate drains away quickly and that steam traps are suitably sized so that condensate doesn't backup onto the heat transfer surfaces.

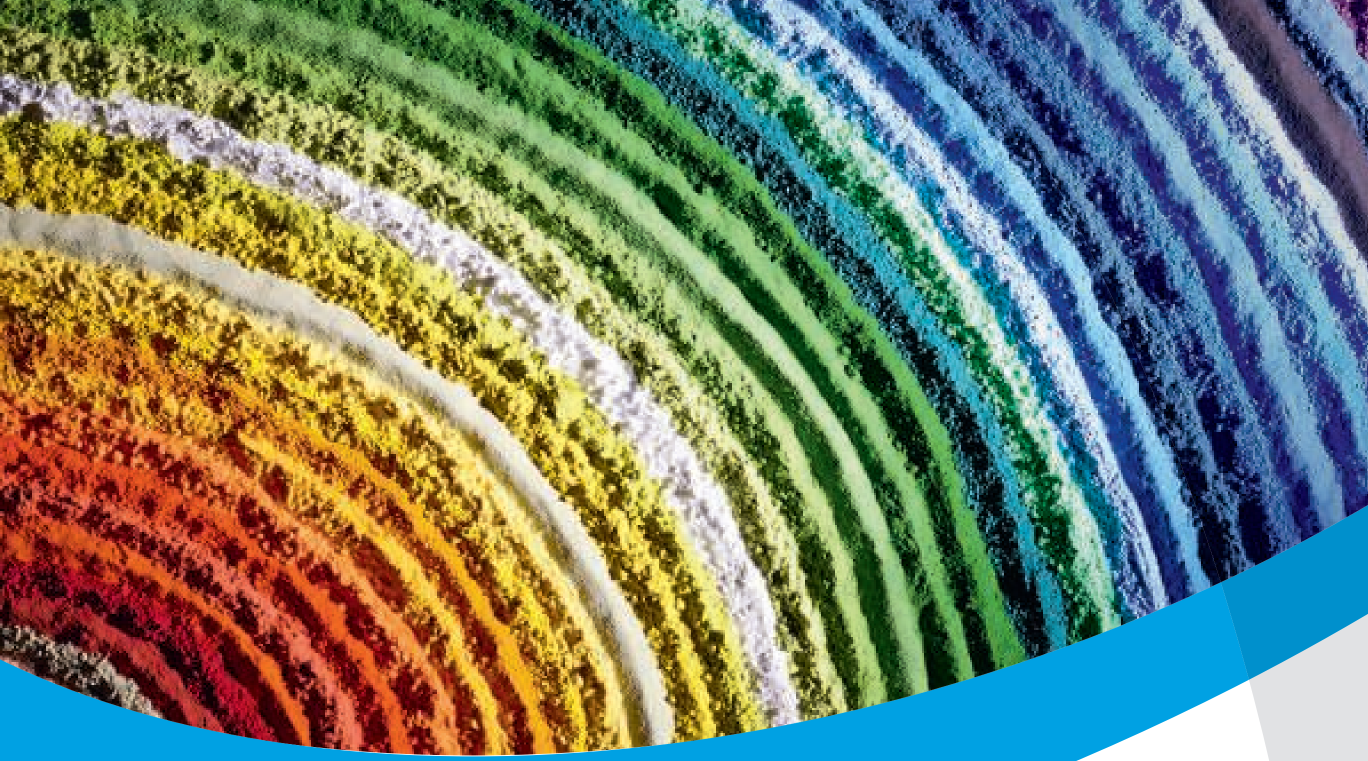
Mike Protheroe
Auckland University of Technology

Editor's note: Thank you for pointing out the errors in the column. Regarding the definition of enthalpy, you are correct — the inclusion of temperature for volume was a typographical error. Regarding the discussion of water films on heat-transfer surfaces, the author was trying to condense a longer explanation, and it was indeed flawed. Thank for you clarifying this. The online version has been updated to reflect these corrections, and can be found at www.chemengonline.com/steam-concepts/.

Making ammonia at milder conditions

There were errors in two chemical formulas in the Chementator article, Making ammonia at milder conditions, (*Chem. Eng.*, November 2016, p. 11). The formulas shown as Ru/CO_2 and Ry/MgO should be Ru/CeO_2 and Ru/MgO. The corrected version of the story can be found online at www.chemengonline.com/making-ammonia-milder-conditions.

Thank you to our long-time reader, Sorab R. Vatcha, consultant from Mountain View, California, for finding the error and bringing it to our attention.



Edited by:
Gerald Ondrey

Algae printing ink has safety and sustainability benefits

Scientists at Living Ink Technologies (Denver, Colo.; www.livinginktechnologies.com) have developed biodegradeable printing ink made from natural cyanobacteria and algae as a sustainable and non-toxic alternative to conventional petroleum-derived printing inks.

Algae-to-ink process

Living Ink's method involves using sunlight and carbon dioxide to grow a variety of algae and cyanobacteria species that are colored by pigmented molecules they produce. For example, green ink comes from chlorophyll produced by the cells, while other strains result in red, yellow and orange inks. Living Ink is also working on a genetically engineered algae strain that can be used for black-colored ink.

To make the inks, algae is grown in controlled containers, then concentrated in a harvesting step (photos). Other plant-based materials are added to the cells to complete the ink formulations, which can then be printed onto paper, cardboard and cotton textiles using conventional printers.

The ink production process is low-cost and simple, because Living Ink uses the whole cell for the ink, rather than extracting pigments from the cells with downstream processing steps, explains Scott Fulbright, the company's co-founder and CEO. For the ink strains, "We needed to find the right combination of pigment color, ability to grow effectively in industrial settings and cell size, which is on the order of 1–5 microns," Fulbright adds.

Living Ink is targeting the packaging ink and stationery markets initially. The company has partnered with algae production company Cellana Inc. (Kailua, Hawaii; www.cellana.com) for high-volume production of algae, and was recently awarded a Small Business Innovation Research (SBIR) grant from the U.S. National Science Foundation to further develop the algae-based inks.

A pilot project to capture rainwater for wine production

A pilot project set up by the University of California Davis (UC Davis; Davis, Calif.; www.ucdavis.edu), GE Water & Process Technologies (Trevose, Pa.; www.gewater.com) and wine-industry service provider Winesecrets LLC (www.winesecrets.com) seeks to capture rainwater for use in wine production, specifically for cleaning the winemaking equipment. Collected rainwater represents an attractive source for water, especially in areas that swing from extreme rainfall to drought, explains GE's sales manager Ryan Hertel. Since rainwater naturally has little alkalinity or mineral content, the water-treatment requirements to achieve comparable purity are much less complex and costly than drawing water from traditional sources, such as wells, aquifers or rivers.

GE provided a reverse-osmosis membrane system and equipment for total-oxygen-content (TOC) analysis to the pilot system, which captures and transports rainwater from the roofs of several UC Davis campus buildings to a holding tank. After subsequent filtration, the water is pumped into 45,000-gal feed tanks for the water-treatment process. "Most of what the membrane technology separates from the rainwater are the dust and particulate matter that settles on the roofs and in the gutters between rainfalls," says Hertel.

UC Davis has been operating the pilot system at its discretion since commissioning was completed in December 2016, reportedly treating about 7,000 gal/d of water. "With the extremely wet and rainy season California has recently experienced, the system has been running a lot," comments Hertel. For the project's next phase, UC Davis plans to livestream the analytics from the treatment system directly over the Internet.

EFFICIENT DISTILLATION

Toyo Engineering Corp. (Toyo; Chiba, www.toyo-eng.co.jp) has completed construction for an energy-saving distillation system used in a project for Maruzen Petrochemical Co. (Tokyo, Japan; www.chemiway.co.jp). This system, tradenamed SuperHIDiC, has achieved an energy saving of over 50% compared with conventional distillation, says Toyo.

The newly installed system was designed with the aim of providing large energy savings for the methyl ethyl ketone (MEK) production plant at Maruzen Petrochemical site in Chiba Prefecture, and is the first commercial application of a SuperHIDiC system.

SuperHIDiC is based on the heat-integrated distillation column (HIDiC) technology, which has been researched for many years by companies, universities and research institutions around the world. Toyo has further refined this technology (*Chem. Eng.*, January, p. 10), in collaboration with the National Institute of Advanced Industrial Science and Technology (AIST; Tokyo, www.aist.go.jp), and plants to expand commercial applications.

SOLAR STEAM

Queen Mary University of London (QMUL; www.qmul.ac.uk) and industry partner Larkfleet Ltd. (Bourne, U.K.; www.larkenergy.co.uk) have received £800,000 (about $98,000) in funding to further develop a concentrated-solar-thermal technology for producing industrial steam. The funding for the two-year project, provided by Innovate UK, the Newton Fund and the National Science and Technology Council in Mexico, will allow an already developed prototype of the solar collec-

(Continues on p. 10)

tor to be improved, to the point of commercialization, at a site in Mexico's Morelos state.

Developed and patented by Larkfleet, the solar collectors use plastic Fresnel lenses instead of conventional glass reflectors. The Fresnel lenses' frame rotates to track the movement of the sun using a fully automatic system, which allows for the concentration of maximum levels of solar radiation on the collector tubes, says the company. The new design is lighter than glass systems, which makes it transportable and potentially easier to manufacture.

The technology is said to be ideal for medium-temperature applications, such as the manufacturing of textiles, chemicals and medicines, for cooking, or any other processes requiring temperatures up to 250°C.

OXIDATION CATALYST

Last month, BASF SE (Ludwigshafen, Germany; www.basf.com) launched Camet ST, a new sulfur-tolerant oxidation catalyst for controlling CO emissions from natural-gas-fired power plants. Camet ST builds on the company's standard Camet oxidation catalyst technology, while also improving the ability of the catalyst to perform in the presence of most forms of sulfur contamination with minimal deactivation. In extended full-scale trials, BASF Camet ST catalyst maintains performance while the competitive catalyst rapidly deactivated, says BASF. Camet ST is currently deployed in several commercial units where sulfur contamination previously resulted in excessive downtime and frequent cleaning.

3-D PRINTING BNNT

Researchers at the Institute for Frontier Materials, Deakin University (Waum Ponds, Australia; www.deakin.edu.au/ifm) have achieved a world-first in successfully 3-D printing a boron nitride nanotube/titanium (BNNT) composite. BNNTs are an advanced new nanomaterial

(Continues on p. 12)

Award to lower costs for flow-battery membranes and reactants

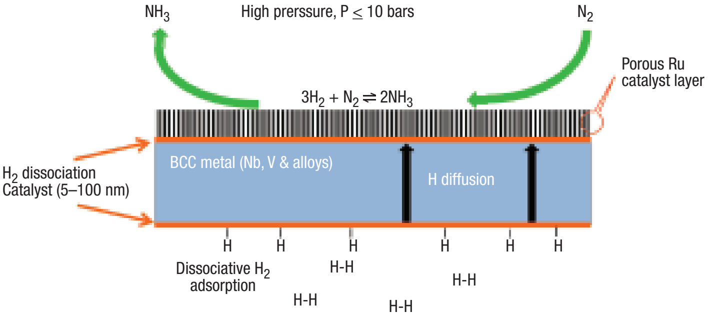
United Technologies Research Center (UTRC; East Hartford, Conn.; www.utrc.utc.com) has been awarded a $2.7-million cooperative research agreement to further develop two technologies aimed at lowering the capital cost of flow batteries for electrical grid-energy storage.

UTRC will lead the U.S. Department of Energy's ARPA-E (Advanced Research Projects Agency – Energy) Integration and Optimization of Novel Ion-Conducting Solids (Ionics) program. Other participants in the Ionics program include Harvard University, Lawrence Berkeley National Laboratory, the University of South Carolina and Advent Technologies.

The research team will seek to build upon UTRC's work on a polar polymer membrane that was originally used in phosphoric-acid fuel cells. The simple, porous ionic separator is doped with sulfuric acid and may obviate the need for more expensive ion-exchange membranes in stationary flow batteries. Such a membrane could lower the cost of that component by a factor of five, says UTRC researcher Michael Perry.

In addition, the award will support development work on lower-cost flow-battery reactants. Currently, most flow-battery reactants are based on vanadium or other transition metals, but the research team will be screening engineered organic molecules for new reactant possibilities. These organic-based reactants, or iron-ligand complexes, could lower flow-battery reactant costs while maintaining electrochemical performance, Perry suggests.

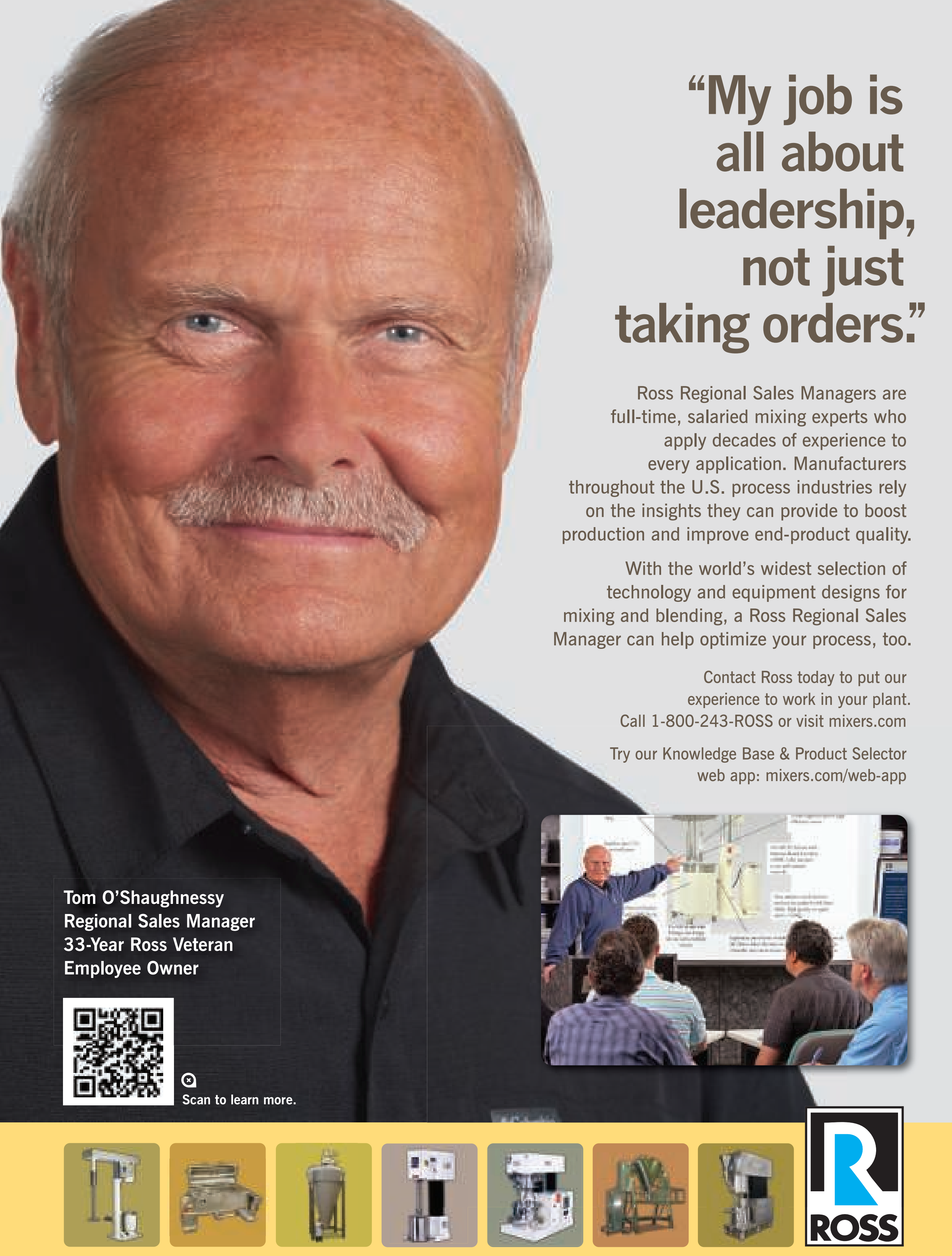
A quest for making ammonia at moderate conditions



Today, ammonia continues to be manufactured by the Haber-Bosch process — an energy-intensive process that operates at high temperatures and pressures (above 400°C and 100 bars). For decades, researchers around the world have been looking for alternative routes to making NH_3, such as those based on electrochemistry, but for now, industrial efforts are predominantly focused on process and catalyst refinements for Haber-Bosch synthesis (see the Newsfront article on pp. 18–21).

A new approach is being investigated by researchers at the Colorado School of Mines (Golden, Co.; www.mines.edu), as part of an "Ideas" project funded by the U.S. Dept. of Energy's ARPA-E program. The 1–1.5-year project aims to demonstrate the feasibility of a low-cost membrane reactor for synthesizing NH_3. The concept uses a hydrogen-permeable metallic membrane to transport atomic hydrogen to the other side of the membrane where the NH_3 synthesis reaction occurs (diagram), explains professor J. Douglas Way, the project coordinator. This makes it possible to decouple the two dissociation reactions of H_2 and N_2, which compete for active sites on the catalyst of conventional Haber-Bosch synthesis, says Way. "We use a niobium or vanadium foil membrane — which is several orders of magnitude less expensive than palladium — coated with an H_2-dissociation catalyst on one side, and a nanostructured N_2-dissociation catalyst on the other side."

In proof-of-concept experiments, a ruthenium catalyst was used for N_2 dissociation, but other materials are being examined as part of the project, says Way. With ruthenium, the researchers have demonstrated NH_3 synthesis at 0.8 bar and 500°C, achieving a higher rate (6×10^{-5} mol/$m^2 \cdot s$) than that achieved by solid-state electrochemical processes, and without the need for supplying an external voltage. Ultimately, the membrane reactor has the potential to increase the NH_3 flux to more than 0.1 mol/$m^2 \cdot s$ by optimizing the N_2-dissociation catalyst, says Way. A patent has been filed on the membrane reactor concept, and Way would welcome hearing from potential industrial partners.



with many unique properties, explains professor Ying (Ian) Chen, chair in Nanotechnology. "They are ultralight, super strong and incredibly resistant to heat," he says.

However, in the 20 years since the material's discovery, it has only been possible to produce it in small amounts, which has seriously limited its practical use in product development. "Our novel and scalable manufacturing process can effectively eliminate this production bottleneck and unleash the real power of BNNTs into the marketplace."

(Continues on p. 13)

Hydronium ions as charge carriers

Scientists at Oregon State University (Corvallis; www.oregonstate.edu) have for the first time demonstrated the use of hydronium ions (H_3O^+) as charge carriers. The research opens another avenue of exploration for high-power, sustainable batteries for stationary power-storage applications.

The research team, led by professor Xiulei Ji, demonstrated that hydronium ions can be reversibly stored in an organic crystalline material, perylenetetracarboxylic dianhydride (PTCDA), which has been used as an electrode in lithium-ion and other batteries. Using dilute sulfuric acid as the electrolyte, the researchers confirmed that the H_3O^+ ions (rather than single protons) were incorporated into the PTCDA crystal lattice, indicating a new chemistry for charge storage, Ji explains.

The researchers also found that H_3O^+ ions diffuse at higher rates than K^+ or Na^+ ions through the electrode structure, suggesting that H_3O^+ has lower migration-activation energy, Ji says. Although the reason for this "low-friction" property is not yet clear, it could eventually give rise to higher-power batteries.

The next stage of research in this area may be to focus on identifying cathode, anode and electrolyte materials for a battery system using H_3O^+ as a charge carrier.

The research was published in a recent issue of *Angewandte Chemie, Intl. ed.*

A less expensive alternative to platinum for water electrolysis

A suitable catalyst for splitting water to make hydrogen must exhibit high H_2-conversion efficiency, excellent durability, and must operate well under low voltage. For an efficient H_2-evolution reaction, the catalyst must be able to trigger proton reduction with minimal overpotential and must have fast kinetics. Currently, the most efficient catalysts in acidic media are platinum-based, because the strength of the Pt–H bond is associated with the fastest reaction rate for the H_2-evolution reaction. However, the Pt-based catalysts are expensive, and are also less stable in an alkaline environment. Catalysts made of inexpensive, non-noble metals corrode rapidly under acidic conditions and operate at very high voltages, which limits productivity.

Now, researchers from Ulsan National Institute of Science and Technology (UNIST; Ulsan, South Korea; www.unist.ac.kr), led by professor Jong-Beom Baek, have developed a ruthenium-based water splitting catalyst that acts almost as effectively as platinum, but is less expensive and is not affected by the water pH.

The UNIST team has synthesized Ru and C_2N, a two-dimensional graphene-like structure, to verify its performance as a water-splitting catalyst. To synthesize the Ru@C_2N catalyst, the team mixed the ruthenium salt ($RuCl_3$) with the monomers that form the C_2N. The catalyst was then produced through reduction and heat-treatment processes.

The researchers used the same processes to produce M@C_2N (M = Co, Ni, Pb, Pt) catalysts. The Ru@C_2N catalyst exhibited the highest performance at the lowest overvoltage. It exhibits high turnover frequencies at 25 mV, as high as Pt, and can be used in any environment.

Remove pollutants from wastewater with this nano-tailored coating

Researchers from the Agency for Science, Technology and Research (A*STAR, Singapore; www.a-star.edu.sg) have developed low-cost, environmentally friendly iron-hydroxide coatings with unique fin-like shapes that absorb large amounts of pollutants, such as dyes, from drinking water, by means of a simple dipping procedure.

Iron hydroxides are being increasingly used instead of conventional activated charcoal (AC), because AC does not easily remove heavy metals and bulky organic compounds from water. Ion hydroxides, on the other hand, can form stable bonds to those pollutants. If iron particles are turned into nanomaterials their, active surface areas are increased, which enhances their chemical adsorption capability. However, it remains difficult to separate iron hydroxide particles from water, which is necessary due to their toxicity.

To overcome this drawback, the researchers synthesizing iron-hydroxide coatings with a hierarchy of structural features, from nano- to micrometer scales, by electrodeposition of aqueous metal ions onto nickel foam at mild voltages. Electron microscopy shows that the coating's nanostructure has elongated, fin-like protrusions, which enable the immobilized nanoparticles to maintain a high active surface area for effective pollutant removal.

The researchers observed that this material safely traps and removes contaminants by immersion in dirty water, and can then be regenerated by means of a simple chemical treatment. For example, they tested the material in water contaminated by a Congo red dye. Within 30 min., the water turned almost colorless, with more than 90% of the dye attached to the special coating. Even though these coatings have some of the highest capacities ever reported, they are only operating at a fraction of their theoretical capacity, the researchers say.

Peltier refrigeration expands into untapped applications

When compared to traditional refrigeration cycles, Peltier (thermoelectric) technology has many benefits, including no moving parts and no hazardous refrigerant chemicals. However, it has not yet been widely adopted into large-scale industrial processes (due to poor efficiency, inability to scale cooling and high costs) until now. Phononic Inc.'s (Durham, N.C.; www.phononic.com) line of Peltier-cooled refrigerators — known as Evolve — have been deployed into the healthcare and life-sciences sectors. The company is also currently investigating the integration of Peltier systems into industrial chilled-water loops. Phononic has built working prototype refrigerators for augmenting chilled-water loops in data-center cooling, and has conducted successful benchtop demonstrations.

Phononic's patented semiconductor chips deliver a large temperature gradient between the hot and cold side while simultaneously pumping a tremendous amount of heat per unit area, says Tony Atti, Phononic founder and CEO. This combination of traits allows for scalability in terms of the amount of heat removed. "As you apply an electric current across a semiconductor chip, one side gets hot and one side gets cold. The application of that coldness determines the commercial efficacy," he explains. However, the key to a robust thermoelectric cooling system is not the chip itself, but the incorporation of the chip into a thermal system that can handle high heat flux with low parasitic losses. "The mechanical system design is just as important as the semiconductor chip itself," Atti comments.

Phononic's thermal design involves combining active and passive heat-transport systems, that when integrated with the advanced semiconductor chips, result in performance metrics that are competitive with compressor-based refrigeration systems. Depending on the application, the Peltier units may consume 10–30% less energy than compressor cycles, says Atti. Phononic's current product offerings include 1.8- and 5.5-ft^3 pharmacy-grade refrigerators, as well as household wine coolers. Later this year, the company plans to release the world's first solid-state freezer.

While BNNTs have a structure similar to carbon nanotubes and share the same extraordinary mechanical properties and thermal conductivity, they are able to withstand double the temperatures (up to 800°C). This superior heat tolerance is critical for surviving the extreme temperatures involved in melting and liquefying powders during the 3-D printing process for metal matrix composites.

BNNTs also have greater chemical stability and electrical insulation properties, the ability to shield against neutron and ultraviolet radiation and to generate electrical current when subjected to mechanical stress.

Deakin has patented its BNNT production technology, which is ready to be scaled up to meet the increasing demand for BNNTs, and plans to build a commercial BNNT pilot plant at its Waurn Ponds campus to produce BNNTs in kilogram quantities.

(Continues on p. 14)

HIGH-THROUGHPUT SCREENING

The number of combinations of substances for coatings formulations is enormous: Even if only ten hardeners, ten binding agents, ten pigments and ten additives are to be taken into account in the development of a coating formulation, this results in 10,000 combinations. And this does not even include variations in proportions. With its new, fully automated plant for testing coating formulations, Evonik Resource Efficiency GmbH (Essen, Germany; www.evonik.com) can now accelerate the search for optimal formulations.

In the first step, the plant dispenses raw materials automatically and formulates them into coatings. In the second step, the substrates are coated with the formulations, dried, and then transported to the test stand. At the test stand, the properties of the formulations are characterized. All steps are carried out automatically according to a precisely defined program that can be reproduced at any time.

The plant consists of 52 elements that are combined in 30 functionalities; each functionality is designed to handle a specific task (for example, to apply a coating formulation to a substrate). The 52 elements are connected by a rail system that goes through all parts of the plant; on the rail systems, containers and substrates are transported via shuttle. In addition, there are 13 robots that carry out various activities, such as loading the shuttle or placing coated substrates in the oven. ❑

New glass electrolytes yield safer, more efficient batteries

A glassy solid-state electrolyte (SSE) developed by researchers from the University of Texas at Austin (UT; www.utexas.edu) will aid in the evolution of safer, longer-lasting rechargeable batteries. A major concern with the use of lithium- and sodium-ion batteries is the formation of dendrites, which can interfere with liquid and crystalline electrolytes, causing battery short-circuits — or worse, explosions and fires. UT's glassy electrolytes are non-flammable and allow for homogenous plating on the cathode and anode, which eliminates the formation of dendrites, say UT researchers Andrew Murchison and Helena Braga. Furthermore, the electrolyte will not oxidize at the voltages needed to charge and discharge the electrodes that are currently available on the market. Simplified battery-cell fabrication, high volumetric energy density and faster rates of recharge are among the other benefits of the glass-electrolyte battery systems.

The new SSE's compatibility with lithium, sodium and potassium enables the use of these alkali metals on anodes, which isn't possible with traditional batteries. Furthermore, the SSE is also universally compatible with off-the-shelf cathodes, enabling its use as a drop-in replacement for liquid electrolytes, according to Murchison and Braga. The SSE is produced via wet chemistry, in a process the team believes could be readily scaled up.

Another potential benefit of batteries employing the new SSEs is the ability to operate at much lower temperatures. "The glass electrolyte has low activation energy for the conduction of the working ions. Therefore, we think it will be able to work at low temperatures," says Murchison. The team has tested cells at temperatures as low as –20°C, and in experiments, the glass electrolyte has performed considerably better than other SSEs at temperatures below 60°C. ■

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Plant Watch

SNC-Lavalin wins EPC contract for ammonia plant in Oman

March 9, 2017 — SNC-Lavalin (Montreal, Que., Canada; www.snclavalin.com) has been awarded a contract by Salalah Methanol Co. LLC (SMC) for the engineering, procurement and construction (EPC) of an anhydrous liquid ammonia plant, including its utilities and offsite infrastructure, in the city of Salalah, located in the Sultanate of Oman. Closing of financing by SMC is expected to occur in June 2017.

Messer opens its largest CO_2-recovery plant

March 8, 2017 — Messer Group (Bad Soden, Germany; www.messergroup.com) recently opened its largest CO_2-recovery plant on the globe. Located in Nanjing, China, the plant has a capacity of 150,000 metric tons per year (m.t./yr). Messer is now recovering a total of 300,000 m.t. of CO_2 from industrial processes per year in China. In addition to the CO_2 recovery plant, Messer is also building a new technology center for gas applications in Nanjing.

Wacker expands silicon-metal capacity at Norwegian production site

March 8, 2017 — Wacker Chemie AG (Munich, Germany; www.wacker.com) is expanding the capacity of its silicon-metal site in Holla, Norway. The company is constructing an additional world-scale plant in Holla, with capital expenditures of around €85 million budgeted for the capacity increase and for expanding local infrastructure. Completion of the plant is expected during the first half of 2019.

Amec Foster Wheeler wins EPC contract from Eni for steam-reforming plant

March 8, 2017 — Amec Foster Wheeler (London, U.K.; www.amecfw.com) was awarded an EPC contract in excess of €50 million by Eni S.p.A. (Rome, Italy; www.eni.com) for a new steam-reforming plant for hydrogen production to be built at Eni's petroleum refinery in Gela, Italy. Amec Foster Wheeler will also support Eni during commissioning, startup and testing. The company's role is expected to be completed in the fourth quarter of 2018.

BASF inaugurates catalyst manufacturing site in Chennai

March 3, 2017 — BASF SE (Ludwigshafen, Germany; www.basf.com) inaugurated its new mobile-emissions-catalysts manufacturing site in Chennai, India. The site includes a new 47,000-m^2 production plant, which replaces an existing BASF plant in Chennai, and is the culmination of a three-year expansion project, which has doubled the company's catalyst manufacturing capacity in India.

Ingleside Ethylene starts up ethylene cracker in Texas

March 1, 2017 — Ingleside Ethylene, LLC, the 50/50 joint venture (JV) between Mexichem S.A.B. de C.V. (Tlalnepantla, Mexico; www.mexichem.com) and OxyChem (Dallas, Tex.; www.oxychem.com), begun operation of its ethylene cracker at OxyChem's Ingleside, Tex site. The cracker has the capacity to produce 550,000 m.t./yr of ethylene.

Arkema to increase PEKK production in France and the U.S.

February 24, 2017 — Arkema (Colombes, France; www.arkema.com) recently doubled the capacity of its poly-ether-ketone-ketone (PEKK) product line in France, and confirms its future investment at its Mobile, Ala. site in a world-scale PEKK plant expected to come onstream in the second half of 2018. These investments will serve the growing needs of the composites and 3-D printing markets.

KBR awarded revamp contract for LG Chem ethylene plant expansion

February 23, 2017 — KBR, Inc. (Houston; www.kbr.com) was awarded a technology licensing and basic engineering design revamp contract by LG Chem (Seoul, South Korea; www.lgchem.com) for its ethylene plant in Daesan, South Korea. KBR will provide services to expand ethylene capacity by 230,000 m.t./yr, resulting in a total capacity of 1,270,000 m.t./yr through the addition of two new furnaces and other system modifications.

CyPlus Idesa inaugurates sodium cyanide plant in Mexico

February 22, 2017 —CyPlus Idesa, a JV between Evonik Industries AG (Essen, Germany; www.evonik.com) and Grupo Idesa S.A. De C.V. (Mexico City, Mexico; www.grupoidesa.com), officially opened its new production plant for sodium cyanide in Veracruz, Mexico. The new plant has capacity to produce 40,000 m.t./yr of sodium cyanide.

World's largest MTO plant starts up in China

February 22, 2017 — Honeywell UOP (Des Plaines, Ill.; www.uop.com) announced that Jiangsu Sailboat Petrochemical Co. started up its methanol-to-olefins (MTO) unit during a 10-day test to confirm successful operation. When the full unit goes online, it will have a production capacity of 833,000 m.t./yr, reportedly making it the largest single-train MTO unit in the world. The facility utilizes UOP's process technology and catalysts.

Mergers & Acquisitions

Evonik and Royal DSM form JV for algae-based omega-3 fatty acids

March 8, 2017 — Royal DSM (Heerlen, the Netherlands; www.dsm.com) and Evonik plan to establish a JV for omega-3 fatty acid produced from natural marine algae. The companies will build a commercial-scale production facility in the U.S. at an existing Evonik site, which is expected to come onstream in 2019. The JV plans to invest around $200 million in the facility.

GE Water & Process Technologies acquired by Suez and CDPQ for $3.4 billion

March 8, 2017 — Suez Environnement S.A. (Paris, France; www.suez-environnement.fr) and Canadian fund manager Caisse de dépôt et placement du Québec (CDPQ) entered into an agreement to acquire GE Water & Process Technologies (Trevose, Pa.; www.gewater.com) from General Electric Co. (GE). The transaction values GE Water at approximately $3.4 billion. CDPQ will hold a 30% stake and Suez will have the remaining 70%.

Cepsa finalizes acquisition of Abengoa biofuels plant

March 6, 2017 — Compañía Española de Petróleos S.A.U. (Cepsa; Madrid, Spain; www.cepsa.com) formalized the €8-million purchase offer for Abengoa's biofuels plant in San Roque, Spain. Abengoa started operations at the plant in 2009, but stopped production in 2015. According to Cepsa, the production unit is due to be fully operative again during 2017.

Synthomer acquires Perstorp Oxo Belgium AB

March 6, 2017 — Perstorp Holding AB (Malmö, Sweden; www.perstorp.com) announced the sale of Perstorp Oxo Belgium AB, Perstorp's additives operations located at its facility at Gent, Belgium, to Synthomer plc (Essex, U.K.; www.synthomer.com). The total consideration for the sale of Perstorp Oxo Belgium is €78 million.

Henkel submits offer to acquire U.S.-based sealant company

March 2, 2017 — Henkel AG & Co. KGaA (Düsseldorf, Germany; www.henkel.com) has entered into exclusive negotiations with GCP Applied Technologies to acquire GCP's Darex Packaging Technologies (Cambridge, Mass.) business for $1.05 billion. Darex is a supplier of high-performance sealants and coatings that generated sales of around $300 million in 2016. Darex operates 20 production sites in 19 countries.

Covestro agrees to sell spray polyurethane-foam business

February 22, 2017 — Covestro (Leverkusen, Germany; www.covestro.com) has agreed to sell its North American spray polyurethane foam business to Accella Polyurethane Systems LLC (www.accellacorp.com). The sale is scheduled to be completed in the second quarter of 2017. Financial terms have not been disclosed. Covestro's spray polyurethane-foam business is located in Spring, Tex. ■

Mary Page Bailey

The Spread of Nitrogen Fertilizers

As price margins tighten, ammonia and urea producers are diversifying their products, while new catalysts and process improvements boost capacities and lower energy consumption

IN BRIEF

DIVERSIFICATION

SAVING ENERGY

NEW CATALYSTS

Haldor Topsoe

FIGURE 1. This facility in Tatarstan, Russia has a nameplate capacity of 2,050 m.t./d of ammonia in a single train, and can be operated to co-produce up to 668 m.t./d of methanol and 1,382 m.t./d of ammonia using Haldor Topsoe's IMAP Ammonia process

Since the start of "shale-gas boom" in 2012, the U.S. has been moving toward becoming an exporter of fertilizer after decades of importing most of its fertilizers. New production plants are beginning to come onstream — something the country has not seen for 35 years.

The startup of new large facilities in major importing nations, like the U.S., Indonesia and a few in Africa has created tremendous price pressure in the ammonia market in the west, says Viswadeb Ganguly, director of Technology, Ammonia & Syngas, KBR, Inc. (Houston; www.kbr.com). Meanwhile, the Chinese export at a very low price has created the same impact in urea processes in Asia, he says. "With quite a few new plants expected to come onstream in 2017 and 2018, it will take some time for prices to improve significantly."

In the last 2–3 years, the market has been characterized by oversupply of urea and declining nitrogen prices, says Merethe Kjul Hoffmann, technology marketing manager, Haldor Topsoe A/S (Lyngby, Denmark; www.topsoe.com). Producers have reacted by diversifying their production, making specialty fertilizer, and revamping plants to improve efficiency and capacity, she says. "Across the board, producers are increasingly interested in sustainable and energy-efficient production, in part driven by stricter environmental regulations. Currently, three new ammonia plants are under construction that will use Topsoe's SCR [selective catalytic reduction] technology that limits the NOx emissions to ultra-low levels below 20 parts per million by volume."

Diversification

New requirements for diversification have increased the interest in Topsoe's IMAP (Integrated Methanol & Ammonia Process) solution for co-producing ammonia and methanol, says Kjul Hoffmann. The IMAP portfolio of processes provide varying ranges of flexibility between ammonia and methanol production tailored to the market needs. The IMAP process combines state-of-the-art NH_3 and methanol technologies in a way that minimize the emissions from the plant, because all CO_2 generated from the process feed is converted to either urea or methanol, and all the process condensate is recycled back to process. Over 24 years, eight IMAP

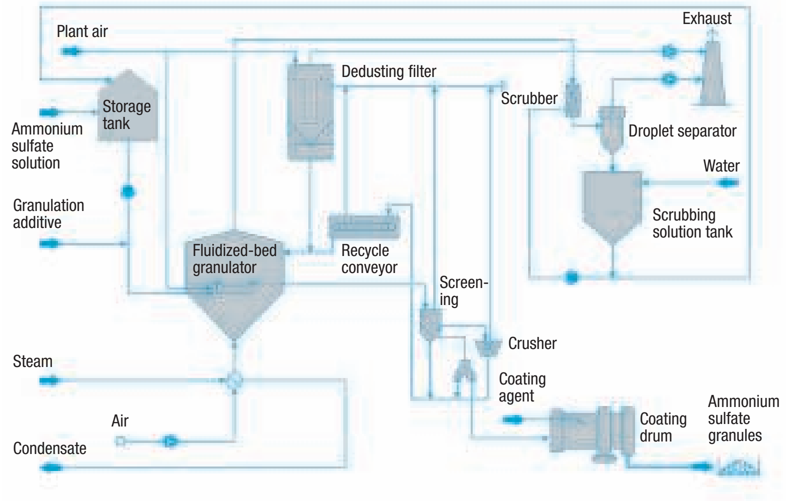

thyssenkrupp Industrial Solutions

FIGURE 2. Launched last month, this patented process produces ammonium sulfate granulate using a fluidized-bed process

plants (both revamps and grassroots plants) have been taken into industrial operation. The world's largest IMAP plant (Figure 1) was commissioned in 2015 in Tatarstan and was the first grassroots fertilizer in the former Soviet Union in decades.

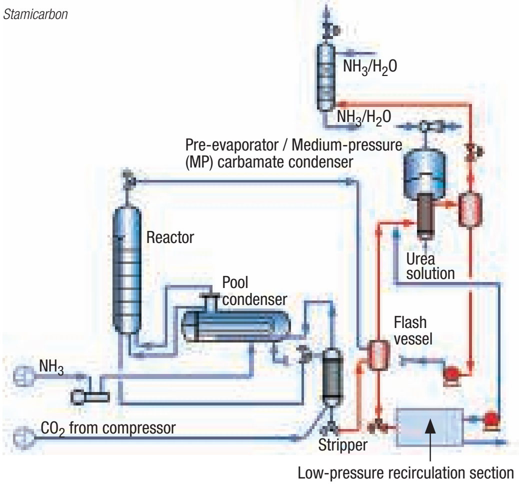
"Margins have become thin for many producers, many of which are looking to sell a product with an edge," says Stephen Zwart, vice president licensing, sales and services, Stamicarbon B.V. (Sittard, the Netherlands; www.stamicarbon.com). That means not just urea, but urea with other additives, such as sulfur or sulfate, micronutrients and others, he says. Also, applications for urea other than fertilizer are emerging, he says.

Ten years ago, 90% of the urea manufactured was used for fertilizer; now it's dropping to 80%, says Zwart. This shows the growth of non-fertilizer applications, he says. For example, the use of urea solutions, known as DEF (diesel exhaust fluids), as deNOx agents for SCR of the exhaust from diesel-engines is now widespread in the U.S., Europe and Brazil, taking off in China, and is expected to grow in other regions as well, such as Southeast Asia and Russia, says Zwart, adding that Stamicarbon has applied technologies in urea plants to produce DEF and improve operating margins.

Stamicarbon's widely used urea fluidized-bed (FB) granulation technology appears to be very suitable for additives to make urea granulate containing sulfur — either as elemental S or as sulfate — and will commission its first urea-ammonium-sulfate (UAS) plant in Russia this year. As an alternative to its FB granulation, the company is also working with pastillation technology in collaboration with Sandvik Process Systems (Fellbach, Germany; www.sandvik.com).

Meanwhile, Shell Thiogro (www.shell.com/sulphur/thiogro) and Uhde Fertilizer Technologies (UFT; www.uhde-fertilizer-technology.com), part of thyssenkrupp Industrial Solutions AG (tkIS; Essen, Germany; www.thyssenkrupp-industrial-solutions.com), established a partnership to integrate UFT's FB granulation technology with Shell's Urea-ES (enhanced sulfur) technology, which was introduced in May 2015. The two companies successfully granulated Shell-developed Urea-ES emulsion in UFT's FB granulation pilot plant in Leuna, Germany. Combining the two technologies will enable the largest urea plants in the world to produce granulated sulfur-containing urea (*Chem. Eng.*, May 2016, p. 8).

The advantages of Shell's technology have been combined with the features of UFT's FB granulation process, where the granulation mode is accretion instead of layering. The elemental sulfur liquor is finely dispersed in the urea melt before the solution is fed to the granulator. Just as in the normal



Stamicarbon

FIGURE 3. This urea process is said to have reduced energy consumption, and also lower investment costs due to a reduction in high-pressure equipment

urea granulation, the Urea-ES particles grow collectively through the solidification of tiny droplets on the seed material. The result is a very hard granule.

Last September, OCP (www.ocpgroup.ma) and Shell signed a strategic agreement regarding the license of Shell Thiogro technology. The Shell Thiogro technology will be installed at OCP's Jorf Lasfar site in Morocco, enabling the company to expand its portfolio by incorporating elemental sulfur into its existing ammonium phosphate, NPKs (nitrogen, phosphorus, potassium) and current sulfur-enhanced products. The technology is expected to be installed and commissioned in 2017.

In a related development, tkIS introduced — at the recent Nitrogen + Syngas conference (February 27– March 2; London, U.K.) — a new granulation process to produce ammonium sulfate granulate. The new plant type produces a sulfur-containing nitrogen fertilizer in granular form from byproduct ammonium sulfate solution. The process offers fertilizer manufacturers major advantages: firstly, much greater cost efficiency compared with conventional processes producing granular $(NH_4)_2SO_4$ from NH_3 and H_2SO_4, and secondly improved spreading and mixing properties compared with crystalline products, says the company.

"Demand for granular ammonium sulfate exists all over the world," explains tkIS's Jens Mathiak, who developed the new process together with his team. "We want to give fertilizer manufacturers the opportunity to convert byproducts into high-quality nitrogen fertilizers, which command premium prices compared with standard products in crystalline form."

The new patented process (Figure 2) starts with ammonium sulfate solution, an industrial byproduct occurring mainly in the production of caprolactam and in coal-fired furnaces. $(NH_4)_2SO_4$ is mixed with an additive, which results in greatly reduced dust formation during granulation and high crushing strength in the end product. In a second step, the liquid mixture is sprayed into a FB granulator and processed into solid granules. The product from the granulator is taken to a screen. Oversize pieces are crushed and returned to the granulator together with undersized particles. Then the product is conveyed to a storage facility. The granules are round and very hard and therefore very resistant to impacts and abrasion.

Following successful laboratory and bench-scale tests, tkIS built a pilot plant in 2016 with an initial production capacity of 500 kg/h. "Here too all the tests have been successful so we can now scale up the process to industrial scale with capacities between 5 and 20 metric tons per hour (m.t./h)," says Mathiak.

Saving energy

Technology providers for ammonia and urea production are continually working to improve capacities through process refinements. "The ammonia technology has continued to improve in energy consumption, unit reliability and ease of operability," says KBR 's Ganguly. KBR's proprietary cryogenic Purifier process can increase plant capacities by up to 50% and, combined with other improvements, can reduce plant energy consumption, says the company.

Extensive concepts have been developed to minimize energy consumption, which are readily available and already proven in installed plants, according to tkIS, and the company says it is continuing to develop its technologies further to make continuous progress on minimizing energy consumption for ammonia production.

Similar efforts are underway for urea production as well, such as Stamicarbon's newly developed Launch Melt Flash Design with improved energy efficiency (Figure 3). With this design, the steam consumption in the urea plant is reduced by about 100 kg/ton of final product by installing a small in-line flashing step between the synthesis loop and the traditional low-pressure stage, explains Zwart. This concept has a better heat integration and reduces the investment cost of the high-pressure (HP) equipment, becoming smaler in size, and some of them dispensed with entirely, he says. Stamicarbon's latest process scheme for the Launch Melt Low Opex design (Figure 4; online version at www.chemengonline.com) allows for steam consumption to be reduced down to around 550 kg/ton of final product.

New catalysts

Catalysts have always played a major role in the production of synthesis gas (syngas; H_2 + CO), the water-shift reaction to make H_2, and the ammonia synthesis itself. Because all of these reactions are energy intensive, it is not surprising that catalyst suppliers continue to improve catalysts to have higher efficiencies, longer lifetimes, lower pressure drops and so on.

Steam-reforming catalysts. Last month, for example, Clariant (Muttenz, Switzerland; www.clariant.com) introduced a new generation of high-performance catalysts for the steam-reforming process in the production of H_2, NH_3 and methanol. The new ReforMax 330 LDP Plus and ReforMax 210 LDP Plus catalysts are designed with a unique and innovative eight-hole flower-like configuration that ensures an extremely low pressure drop in the tubular reforming reactor while offering outstanding catalyst activity and selectivity, says the company.

The shape of the catalyst, used for primary reforming, plays a key role in the catalyst's activity, heat transfer, pressure drop and physical strength.

The new ReforMax LDP Plus catalysts offer optimized shape parameters. Compared to its ten-hole predecessor, the new eight-hole “floral” design of ReforMax LDP Plus allows a pressure-drop decrease of up to 20%, says the company. This allows plant operators to significantly reduce energy costs or increase the reformer gas throughput (or both) in existing plants.

Moreover, the new catalysts retain the very high geometric surface area of Clariant's well-known ReforMax LDP ten-hole catalyst series, thus maintaining high activity for the steam reforming reaction. Heat transfer is improved thanks to increased hole diameters. Particularly when used together, ReforMax 330 LDP Plus and ReforMax 210 LDP Plus effectively suppress carbon formation in the process, adds Clariant.

Another way to reduce pressure drop in steam reformers is the Catacel SSR catalyst from Johnson Matthey plc (JM; London, U.K.; www.matthey.com). In the Catacel SSR system (*Chem. Eng.*, March 2010. p. 11), which JM acquired in 2014, alloy strip is formed into engineered supports called fans, which are coated with a nickel-based steam-reforming catalyst. The fans are stacked inside of the reformer tubes. This design offers many advantages over traditional ceramic pellets, and provides lower pressure drop, high heat transfer and high activity, says the company. Introduced several years ago, Catacel SSR is said to represent the single biggest step forward when it comes to the development of catalyst shape. Compared to standard pellets, Catacel SSR leads to a 20% decrease in pressure drop. “The value of increased efficiency when exchanging an old catalyst with a new optimized one can normally pay for the optimized catalyst charge within 1–2 yr of operation, says the company.

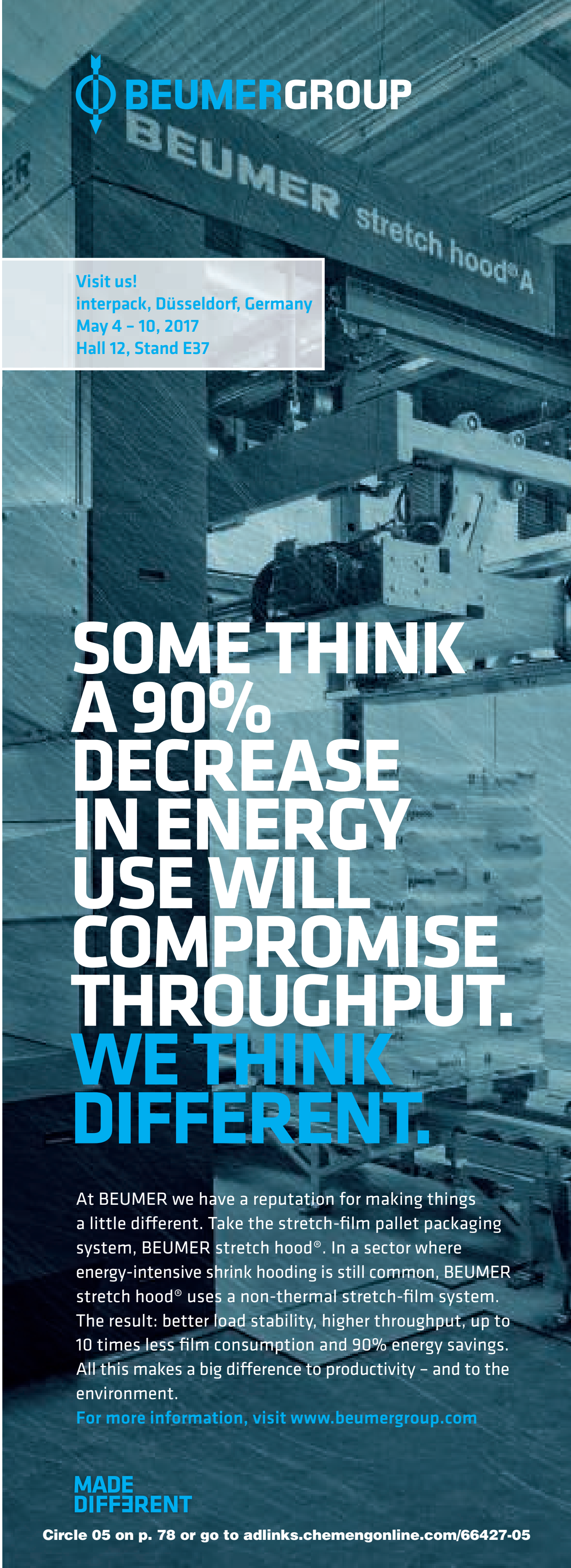
Shift catalysts. In February 2016, Clariant introduced the Low Temperature Shift (LTS) chloride guard ShiftGuard 200 into the market. ShiftGuard 200 effectively adsorbs and retains chlorides — a typical catalyst poison in an NH_3 or H_2 plant — so that the downstream main LTS catalyst, such as Clariant’s ShiftMax 217, is fully protected.

In September 2014, Clariant also launched ShiftMax 120 HCF, an enhanced high-temperature shift (HTS) catalyst that avoids health and safety risks in NH_3 or H_2 production because it contains essentially no hexavalent chromium (Cr^{6+}), says the company (for more details, see *Chem. Eng.*, October 2014, p. 14)

Last year, Topsoe launched a new high temperature shift catalyst SK-501 Flex, which uses a promoted magnesium/aluminum spinel structure instead of the traditional iron/chromium structure. It removes the steam-to-carbon ratio bottleneck and lets NH_3 producers save on feedstock and energy or boost production by up to 5% in their existing set-up. The new catalyst also eliminates the risks related to Cr^{6+}, both to the environment and plant personnel, says Topsoe’s Kjul Hoffmann.

NH_3 synthesis. In 2014, Topsoe introduced the KM 111, an NH_3 synthesis catalyst optimized for the lower beds of the converter with a high concentration of NH_3. KM 111 lifts the performance of the NH_3-synthesis loop significantly and improves energy efficiency, says the company. ■

Gerald Ondrey



The Future of Safety Sensors is Here Now

Wearable sensors enhance CPI worker safety and help close the approaching skills gap

IN BRIEF

- THE FUTURE OF SAFETY IS HERE TODAY
- THE SUM OF THE PARTS
- COLLECTING DATA
- THE FUTURE OF WEARABLE SENSORS
- PROCESS SENSORS USED FOR SAFETY APPLICATIONS

For many years, sensors have been used in the chemical process industries (CPI) for many activities, including monitoring the health and safety of employees. In these applications, sensors were especially helpful when used to accumulate data on workers, identify potential exposures to hazardous chemicals and to monitor confined space entry and dangerous inspections. However, many of the sensors that were used for these applications were large and had to be carried by hand or placed at a specific point within the plant or process line. More recently, the advent of smaller, advanced sensor technologies and the ability to locate them in wearable items, such as helmets, smart watches and smart glasses, and connect them to today's powerful communications infrastructures, all contribute to enhance the safety of CPI employees.

American Industrial Hygiene Association

FIGURE 1. Smaller, wireless sensors have the potential to change the health and safety paradigm regarding collection of employee exposure data

"With the advent of smaller sensors and microprocessors, monitors are becoming compact and wearable. They are wireless, so it is no longer necessary to have wires that interfere with an employee's work, and they can transmit the information to the Internet of Things or the cloud so that data can be downloaded into a computer and analyzed in real time," explains Russell Hayward, managing director of Strategic & Technical Initiatives with the American Industrial Hygiene Association (AIHA; Falls Church, Va.; www.aiha.org). "Smaller, wireless sensors have the potential to change the health and safety paradigm regarding collection of employee exposure data" (Figure 1).

For instance, Hayward says, with the proper procedures and processes in place, wearable sensors can now be placed on all operators in a particular chemical plant unit and set to alert workers when they enter an area of excessive exposure to a particular substance. "When the alarm triggers, the exposure is investigated and the data points are captured in a central repository and analyzed. It can then be determined when, where and why the alarm triggered and the situation can be corrected in a virtually realtime manner," he says. "Prior to advanced sensor technology, we didn't have the opportunity to monitor all operators and identify the areas of risk that operators were encountering on a task-by-task basis."

Another game changer is the ability to place arrays of these advanced, miniaturized sensors around operating units and along facility fence lines and, during emergency response situations, link them to a communications infrastructure and provide virtually realtime data analysis. "In these situations, the data can be used to make emergency response decisions."

The future of safety is here today

If this safety-enhancing sensor technology, coupled with wearable devices such as helmets, smart watches and smart glasses, seems like something that might be seen in the future, think again. It's here now and is slowly beginning to make its way into the world of industrial processes.

For instance, Honeywell (Morris Plains, N.J.; www.honeywell.com) offers its Connected Worker solution, which provides safety intelligence to reduce worker injuries and prevent loss of life from "man-down" scenarios. The solution collects and provides sensor fusion, which refers to data collected from a variety of sensors on a worker that are compiled to provide an accurate picture of what that worker is experiencing. The solution monitors workers for toxic gas exposure, breathing, heart rate, posture and motion. The resulting data and actionable intelligence are displayed remotely on a visual, cloud-based dashboard, giving plant managers and incident commanders the information needed to better anticipate unsafe conditions and prevent potential "man-down" scenarios that could threaten worker safety.

Upskill

FIGURE 2. The benefit of wearable technology, such as smart glasses, is that their hands are now free so workers can keep their hands and eyes on the tasks at hand. This provides increased productivity, improved safety and better efficiency in activities throughout the facility and in hazardous situations

"Employers are always trying to operate a safer environment, so we tried to create a solution that would help them with that. It can help protect the lone worker — those in hazardous areas or remote locations — because it uses the sensors and the connectiv-

PROCESS SENSORS USED FOR SAFETY APPLICATIONS

If you’re in the market to increase facility and employee safety but aren’t ready to don wearable technologies just yet, experts suggest looking outside the realm of safety sensors and into sensors that weren’t traditionally used for safety applications. Sensors such as those used for level and leak detection and tunable diode lasers (TDLs) are currently being employed in chemical applications to avoid accidents and prevent processes from going out of control, keeping plant and personnel safer than ever before.

“What we’re seeing is the application of sensors that were traditionally used for other applications being applied and related to the safety of not only the process, but the people around the process, as well,” says Gene Cammack, director, System Product Marketing with Yokogawa Corp. of America (Sugar Land, Texas; www.yokogawa.com). “For example, we are applying TDL analyzers into a lot of safety applications because it gives an open path that scans all the way across the boiler to look for specific gases that may create problems with safety,” he says. “We hadn’t really done that before, but it’s an excellent application and now the TDL technology is having a moment as a safety-related sensor, rather just in its traditional use for measuring the content of the process.”

Because the TDL technology shoots a beam across a space and scans for specific gases, it is possible to set it to scan for gases such as oxygen, carbon monoxide and hydrocarbons in a boiler. “The sensor technology allows sight into different areas of a boiler that are more susceptible to buildups of gases, which can create fire or explosion hazards. “Many of these areas couldn’t be measured before, but because TDLs are a non-contact technology, the sensors don’t have to be embedded into the gas stream. So instead of using a point sensor, the TDL provides a broader look at what’s happening inside, all the way across the boiler, making it much more advantageous to sensing the safety level in boiler, cracker or refinery application where explosions occur due to offgas in the electrostatic precipitators.

He adds that another bonus of the TDL technology is that the information is provided in realtime, which traditional safety sensors for these applications can’t provide. “This is why it’s a big deal in the safety world.”

Tina Hull, product engineering-safety, with Omron Corp. (Hoffman Estates, Ill.; www.omron.com) sees a similar trend. “We are learning to apply a lot of non-safety sensors to detect activities or events that could potentially send a human into a hazardous area or situation, so by applying more advanced sensors for things like leak detection and motion, we are providing more benefits than safety sensors alone.”

For example, she says, detecting when a leak begins and correcting it quickly can reduce the probability of a slip or fall. While the sensor’s primary purpose is detecting leaks on applications such as measuring baths, pipe joints for chemical tanks or fluid levels, the safety application is in correcting the issue immediately and reducing the probability of unnecessary future access into the hazard area.

And, non-traditional sensors can be used in conjunction with safety sensors for further improvements. For example, when using machine automation controllers, which integrate motion, robotics, safety vision and sensing along with Omron’s SQL database, a date and time stamp can be tracked. “This information can be used to look for trends and lead to design improvements,” says Hull. “For example, the liquid-leak sensor may detect leakage on a cleaning device at a consistent time, such as 4:00 PM, and the safety devices record that the interlocking safety switch is activated within 30 minutes of detection. Activating the interlocking safety device puts the machine into a safe state. The data could be a warning that a change elsewhere in the system might be needed,” she says. “Perhaps there is a heating element too close to the cleaning station that either needs to be placed at another location or it needs to be insulated. This type of change would then reduce the leakage pattern, and thus reduce the operator’s need to access the hazardous area, and even increase production.”

In addition, safety is further promoted because the devices are becoming more reliable thanks to self diagnostics, says William Sholette, level product business manager for Endress+Hauser, Northeast, (Greenwood, Ind.; www.us.endress.com). “There are more diagnostics inside the unit, such as watchdog circuits to make sure the microprocessors are all working and communicating with each other,” he says. “This is constantly done in the background and is monitored all the time so, if it fails or gets into a fault condition, a notification is sent to alert users that something is wrong.”

He adds that some companies, such as E+H, take self diagnostics a step further in some sensors, such as the company’s FTL 81-point level switch, and add a square wave signal onto the current so that it is constantly modulating. “The unit makes sure it can actually change current by looking at the square signal. If it is modulating, the current can obviously change, and therefore, it will change in a situation where it needs to go into a fault condition,” he explains. “We are trying to make these units, especially those used in personnel and process safety, more reliable and more responsible for themselves so they will reliably signal if there’s a problem that needs attention.”

“It’s an interesting and evolving area as we try to figure out how to keep people and processes safe, especially as we scale back on human resources and make things more automated,” notes Cammack. “We have to ensure that safety is built into the systems and ensure that we maintain the sensors and other parts of the functioning safety system. Everyone is trying to do more with less, but they don’t want to do it with less safety.” ❑

ity element to alert the worker that he or she may be in a dangerous situation and also gives an alert to someone if the worker is in trouble. It also provides situational awareness in that the sensors are consistently measuring exposure limits and warning the workers of danger. There’s a third benefit around worker productivity, as the connectivity can be used to provide information to the workers with instructions about work flow,” says Prabhu Soundarrajan, Connected Worker leader, with Honeywell.

Upskill, formerly known as APX Labs, (Herndon, Va.; www.upskill.io) provides its Skyline platform, which powers enterprise wearables that use sensor technology to connect workers to realtime information and to each other (Figure 2). “The fundamental purpose is connecting, keeping safe and providing information to the hands-on workforce as they do their work,” says Jay Kim, chief strategy officer with Upskill. “The benefit of wearable technology, such as smart glasses, is that their hands are now free so workers can keep their hands and eyes on the tasks at hand. This provides increased productivity, improved safety and better efficiency in activities throughout the facility and in hazardous situations.”

Further, says Kim, the technology provides critical sensor nodes within the visual factory. “Glasses and wearables go everywhere hands-on employees go, so a tremendous amount of data is generated by these operators,” he says. “Those data go back into the system to make data analysts and workers even smarter.”

In addition, the wearables allow information sharing. Shared information could include the actions and steps of safety procedures, such as lock out/tag out. Another safety-enhancing example might be the use of what Kim refers to as “a simple see-what-I-see” scenario in which the number of people sent into a hazardous area can be reduced because

Vandrico Solutions

FIGURE 3. Vandrico Solutions is exploring the use of networked sensors coupled with software solutions and electronic technologies to increase the safety of miners

remotely located experts could provide "over-the-shoulder" coaching to an individual completing tasks in a dangerous area using a front-facing camera on the smart glasses, rather than sending the entire team in to complete the activity.

And, Vandrico Solutions (Vancouver, Canada; www.vandrico.com) is exploring the use of networked sensors coupled with software solutions and electronic technologies to increase the safety of miners (Figure 3). One of the potential uses here would be to use sensors that are able to determine shifts in rock and seismic activity, which are linked to software and wearable devices and used to send realtime alerts to workers in an automated way. "In mining, it's often difficult to get signal propagation, so one of the theories we have is that wearable technology, such as helmets, smart watches and head-mounted displays, make sense because it goes on the body, which helps determine the realtime location of the worker, if needed, and gets the worker's attention," says Gonzalo Tuleda, CEO with Vandrico. "With the prolific use of smartphones, we all have notification fatigue and tend to ignore a lot of notifications. But a vibration on the wrist or a flashing light under a hard hat will get the worker's attention when needed. By combining the sensors that are in the mine with a sensor on wearable technology, you can provide realtime safety alerts in an automated way."

The sum of the parts

Advanced safety solutions such as these are dependent upon several system components. According to Honeywell's Soundarrajan, in most solutions, advanced sensors are used to measure the hazard, such as a chemical parameter, gas or chemical substance. That data gets communicated from point A to point B via a communications infrastructure,

such as a bluetooth transmitter that takes the information to a cell phone or wearable device or a WiFi network that takes it to a control room or the cloud for storage. The communication infrastructure may also include location technology to track the location of the employee and sensor. And the third component, according to Vandrico's Tuleda, is software. "Software is needed to help translate information from the sensors and the platform with which the sensor is communicating. The software should also have a logic component that is capable of analyzing the data and making realtime decisions about sending out alerts if set limits are surpassed or an incident is about to occur," says Tuleda.

In addition, software connects the workers and the wearables to the IT infrastructure so they can access the information they need, and also has the capability to connect workers to the Internet of Things or WiFi network to provide situational awareness, says Kim. "Software platforms connect the human interface to the rest of the system," he says.

Collecting the data

Prior to the use of advanced sensors, sophisticated software might not have been as important because there wasn't as much data. "We were happy to have one or two data points per shift on an operator in a process unit," says AIHA's Hayward. "But with the advancement of today's direct-read instruments, it may be possible to capture one, two, six or more data points per second. With that much data, it is becoming necessary to identify or develop tools, techniques and knowledge to analyze large data sets in order to make good, useful, scientifically based decisions."

Honeywell's Soundarrajan calls this "data with context." This is where the intelligence of connecting data collected by the sensors with software comes into play. "We are developing software and successfully implementing software architectures that are capable of collecting data and reporting exceptions."

Today's software is capable of collecting data, storing and analyzing it and then using that information to predict an incident before it happens. "The ability to alert users on their mobile or wearable devices notifies them and allows them to react to the situation quickly, which has the potential to save lives and millions of dollars," says Soundarrajan.

He adds that other software advancements allow the software to do a good sum of the work traditionally done by industrial hygienists. "Today in industry, the data are often in silos, sitting in different systems, but using software that brings it all together — from the data from the sensor level that is associated with a worker all the way to the enterprise systems. This means the experts don't have to do the crunching of data. Typically, hygienists spend at least 8 to 10 hours a week with Excel sheets, but today's technology can reduce the burden of analysis. Regular reports are now available on demand."

In addition, those data are available if an incident has occurred or if a regulatory agency requests a report. "Hygienists can pull that report in seconds, rather than crunching numbers for 10 hours," he explains.

In addition, software and stored data can be used to lessen the burden of the ever-growing skills gap in the process industry, says Upskill's Kim. "One of the biggest themes we see in the industrial workforce is that there is a potential skills gap. The baby boomers have become the real domain experts, but as they are heading to retirement and millennials are coming in to replace the workforce, the facilities are at risk of having a tremendous loss of knowledge that wasn't captured in their digital systems," explains Kim. "Equipping workers with wearable, connected devices enables easier capturing and sharing of that tribal knowledge and information. In some cases, it can be as simple as recording a video of a certain process and associating it with a specific workflow. Once that is stored in the system, it can then be made available to trainees and employees who may not know how to complete the task. This has the potential to eliminate the expected knowledge and skills gap."

Vandrico's Tuleda agrees and says it is possible to take it a step further in the future: "We have the opportunity to capture the knowledge that the experienced workers have and the decisions they make given the data they are presented. We can capture those data and use machine learning algorithms to train artificial intelligences, which allows us to make a giant step toward the automated decision component of industrial and process operations," he says. "The things smart sensors do reliably allows us to tap into the knowledge of trained, experienced people and learn from their decisions what works and what doesn't and then transmit that information in both directions — into the system and into the hands of the worker in the field."

The future of wearable sensors

Right now, such advanced technologies are in the early adoption stage with larger enterprises and, within those businesses, are mostly being employed in critical applications. However, early adopters are beginning to see enough significant increases in efficiency and safety that many are moving the technology into full-scale operational settings. "We are seeing not only early adopters making excellent progress with successfully implementing this technology into real operational settings, but we are also seeing them doing this faster than they have over the past five or six years. The technology is getting into the mainstream mindset of operational leaders in larger enterprises, which means it will likely begin to accelerate with new customers at a rate that is faster than it has been in the past," says Kim.

Honeywell's Soundarrajan adds that the chemical industry is showing interest because they are constantly pushing the envelope on safety. "Sensing and connected systems are an excellent way to share knowledge and improve safety," he says. "And for the chemical industry, which is a risky environment, this type of connected sensing technology helps make their world a much safer place." ■

Joy LePree


Flow
2836.72 SCFH

Pipes, Tubes and Fittings

Esco Tool

This pipe-milling end-prep tool is for beveling boiler tubes

The Esco Wart Millhog (photo) is a pipe-beveling machine that is designed for making repairs to waterwall boiler tubes. It features a rigid-inner-dia. clamping system, and uses titanium-nitride-coated cutter blades to machine carbon steel or highly alloyed tubes without the need for cutting fluids. The machine is able to bevel, face and bore simultaneously, with relatively simple tooling changes, says the company, and it is designed to machine water-boiler tubes with inner dia. from 2 to 3 in. The machine is sealed to prevent debris from entering the tool. It rigidly attaches to the tube with clamps that fit the inner dia. and expand on the mandrel using a self-centering, draw-rod assembly and attached wrenches. The machine is available with both pneumatic and electric motors. It is available for purchase or for rent. — *Esco Tool, Holliston, Mass.*
www.escotool.com

Michael Smith Engineers

Drum pumps now offer a broader choice of tubes

These high-performance sealless drum pumps (photo) are designed for transferring liquids from intermediate bulk containers (IBCs). The pumps are now offered with a choice of six tube lengths. The newest tube length option (137 cm; 54 in.) is available in several materials (two polypropylene versions, a polyvinylidene fluoride option, and a stainless steel version). This variety of wetted materials ensures resistance to the widest range of aggressive liquids and therefore maximum application flexibility, says the company. The other key features of the PF Series drum pumps include a sealless design that is said to improve reliability, a double-suction impeller that provides high-flow and high-head capability (flows to 151 L/min., and heads to 24 m), and easy disassembly for cleaning. The tubes are interchangeable with different motor options. — *Michael Smith Engineers Ltd., Sheerwater, U.K.*
www.michael-smith-engineers.co.uk

Parker Hannifin

Flat-faced seal replaces elastomeric O-rings

The Parker Seal-Lok Xtreme line of flat-face seal fittings (photo) was recently awarded a patent for its newest addition — a stainless-steel, flat-face sealing ring that is designed for use with tube fittings in extreme- (high- and low-) temperature applications. The innovative metal seal features a unique trap-style seal with three retainer clips that extend into the existing fitting's O-ring groove and prevent the seal from falling out during assembly or reassembly. It is said to provide an alternative to elastomeric O-rings in traditional O-ring face-seal fittings that are not able to meet high- or low-temperature or chemical compatibility requirements of certain applications. These metal seals can be used in applications from –328 to 1,200°F (–200 to 650°C), while elastomeric O-rings seals are limited to a temperature range of –65°F to 450°F (–54°C to 232°C). This seal is designed for liquefied natural gas (LNG) storage and fueling systems, combustion turbines, cryogenic applications, and other demanding CPI applications, according to the manufacturer. — *Parker Hannifin Corp., Columbus, Ohio*
www.parker.com

Silicone hose is compatible with many clean applications

The APSW high-purity silicone suction hose (photo) now has a National Sanitation Foundation listing (NSF-51). The hose is designed for ultrapure water transfer, food processing, beverage conveyance, washdown stations, skid systems and other applications in the pharmaceutical, biopharmaceutical, bioprocessing, chemical and cosmetics industries. The APSW hose begins with a liner of low-volatile-grade, platinum-cured silicone. This liner is then slipped onto a mandrel and wrapped with four plies of polyester mesh fabric, 316 stainless reinforcing wire and additional silicone. It is then cured into a smooth and uniform hose that can handle both suction and discharge applications. The hose is available in

Note: For more information, circle the 3-digit number on p. 78, or use the website designation.

New Age Industries

smooth and convoluted-outer-diameter styles, can withstand temperatures from –100 to 350°F, and may be sterilized using several different methods. — *New Age Industries, AdvantaPure Div., Southampton, Pa.*
www.newageindustries.com

Break-away coupling improves safety during tanker loading

Emco Wheaton

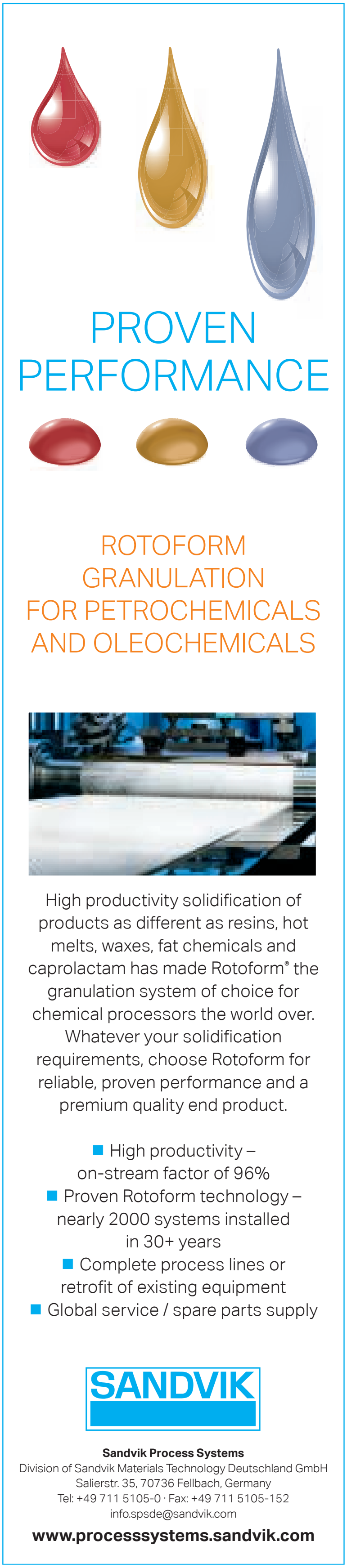
The TODO TTMA 4-in. Aluminum Break-Away Coupling (photo) is designed to reduce the potential for damage and spillage and product loss during the loading and unloading of road tankers. The device has been adapted for bottom-loading-arm applications, and it can be used on new loading arms, or retrofitted into existing ones that currently use the same type of TTMA flange in their construction. It consists of two identical halves joined together by a series of 3-mm break pins allowing the device to be configured to separate at a defined load. Should an excessive load be placed on the loading arms, the pins automatically close the internal valves, to protect the driver and the environment. The device can then be reset onsite. — *Emco Wheaton USA, Houston*
www.emcowheaton.com

Silicone masterbatch improves polyethylene processability

The MB25-502 silicone masterbatch provides processability advantages for polyethylene (PE) compounds that are highly mineral-filled (to improve flame retardancy) and used in wire and cable insulation and jacketing applications, says the company. By reducing screw torque, die buildup and die pressure, the material can improve extrusion throughput by up to 110% compared to PE compounds without a processing additive. The MB25-502 masterbatch enables wire and cable customers to maximize productivity without paying a premium for unneeded functionality, says the company. It raises the throughput of PE compounds with mineral loadings up to 70 wt.%, by reducing screw torque by as much as 30%. Lower torque, in turn, speeds production and cuts energy costs. The silicone masterbatch also avoids buildup on the die (and increased die pressure) that would require frequent interruptions for cleaning. The product is compatible with low-density PE, linear low-density PE, and cross-linked PE, and it is supplied globally as free-flowing pellets. — *Dow Corning, Midland, Mich.*
www.dowcorning.com

Fittings now have Type 316 stainless-steel options

The T&B Liquidtight Systems family of stainless-steel fittings (photo) for stainless-steel flexible conduit has added a new addition: SAE Type 316 stainless steel, a marine-grade stainless steel with enhanced resistance to galvanic corrosion in food-and-beverage applications and other particularly corrosive settings. The design of these fittings has also been updated for improved protection against liquid ingress. New features include the dome-shaped gland nut that is unique to this family of products, says the company, which prevents liquids from pooling in the electric box. And, the SafeEdge ground cone securely attaches to the conduit for improved pullout performance that exceeds UL requirements, says the company. Sizes range from 3/8 to 2 in., and they are available in 45-deg, 90-deg and straight configurations. — *Thomas & Betts, Memphis, Tenn.*
www.tnb.com



Thomas & Betts

Viega

Isolation joint improves safety of flanged pipe connections

The ElectroJoint monolithic isolation joint replaces flanged-joint connections with a weld-in-place connection, which is said to reduce human error, reduce corrosion and increase safety. The isolation joint replaces bolts, sleeves and washers with a coated, sealed cathodic-protection system to eliminate corrosion, is secured with two butt welds, and is especially well-suited for smaller-diameter pipes, including those used in gas-distribution lines, says the company. — *Garlock Pipeline Technologies, Houston*
www.garlock.com

Cold-press connections for pipe installation are versatile

MegaPress and MegaPressG fittings, such as reducers and unions (photo), are easy to use, flameless and provide secure, watertight and airtight connections. These fittings are FM-compliant for fire sprinkler applications and ASME B31 compliant for pressure and boiler piping, and they are CSA LC4 compliant for fuel -gas and oil applications. The design reduces buildup of joining materials, exposed threads or tarnish, creating a clean, professional appearance, says the company. Since the system of fittings is flameless, it is safer than traditional systems and eliminates the need for hot-work permits. The system requires no welding rods or cutting oils. It can be installed under flow conditions, making it ideal for maintenance or emergency repair jobs and reducing costly downtime, says the company. More than 200 MegaPress fittings, including elbows, couplings, reducers, tees, reducing tees, threaded adapters, caps, flanges and unions, are available in sizes from 0.5 to 2 in. — *Viega LLC, Wichita, Kan.*
www.viega.us

Single-use tube assemblies are designed for bioprocessing

The Puresu family of single-use tubing assemblies provides a broad range of customizable configurations and components, which can be ordered with no minimum quantity. Full traceability of each component is maintained throughout the assembly process and is provided with detailed documentation. A range of materials are available, as are numerous associated clamps, valves, adapters and connectors. This ensures that these single-use tube assemblies are ready to use upon delivery, and meet the exacting requirements by cGMP manufacturing and validation standards, says the company. — *Watson-Marlow Tubing, BioPure Div., Wilmington, Mass.*
www.wmftg.com

These hoses are designed to deliver liquid cryogens

This company offers a variety of cryogenic transfer hoses, to ensure the safe and efficient transfer of liquid crygogens, including liquid helium, liquid nitrogen, liquid argon and liquid oxygen. The hose options range from simple, non-jacketed flex lines to vacuum-encased systems, for use in industrial settings, laboratory settings or as an instrumentation accessory. Users can match the materials of construction to the cryogenic liquid being transferred, and choose from many options, including rigid versus flexible hoses, and may consider uninsulated, vacuum-insulated or foam-insulated designs, to suit the needs of the application. The company also offers a range of components, such as, tanks, valves, bayonets, and more, as well as custom system design. — *Cryofab, Kenilworth, N.J.*
www.cryofab.com

Handy tool lets users design pipe transits in the field

Roxtec Transit Designer 4.0 lets designers and engineers envision and design pipe and cable routing in the field, and it provides full documentation (ranging from certification to installation instructions). The latest improvements to the tool include a product selector that helps users identify the right sealing solution for each applications. — *Roxtec International AB, Karlskrona, Sweden*
www.roxtec.com ■

Suzanne Shelley

New Products

A compact compressor for H_2-refueling stations

This company has started marketing the HyAC mini-A (photo), an all-in-one, compact compressor package for stationary hydrogen refueling stations designed for use in the U.S. The HyAC mini-A consists of a high-pressure H_2 compressor and a refrigerator sold together with a high-pressure storage tank unit and a dispenser as a set. The HyAC mini-A has a footprint 10% smaller than the HyAC mini sold in Japan since 2014. The HyAC series consists of two models: a single-unit, high-pressure H_2 compressor, named HyAC, and a compact package consisting of the hydrogen compressor and other major equipment called the HyAC mini. To meet the U.S. fueling protocol, the filling pressure of the HyAC mini-A was raised to 87.5 MPa. The HyAC mini-A is equipped with a remote monitoring system, and also includes compact micro-channel heat exchangers called DCHE. — *Kobe Steel, Ltd., Tokyo, Japan*

www.kobelco.com

This range of diaphragm pumps is extended

Launched at this month's Hannover Fair (April 24–28), the hydraulic diaphragm pump Hydro/ 4 (photo) supplements this company's Hydro/ 2 and Hydro/ 3 types, thereby extending the application range to higher capacities. The pump covers the capacity range from 130 to 1,680 L/h at 7 to 25 bars absolute. The Hydro/ 4 is equipped with a polytetrafluoroethylene (PTFE) multi-layer metering diaphragm. The multi-layer diaphragm ensures safe and leakage-free metering. With material options including stainless steel, polyvinylidene difluoride (PVDF) and Hastelloy C, the Hydro/ 4 can be used universally in numerous applications. As standard, all pump types are equipped with a fixed-setting pressure-relief valve that is integrated into the hydraulics and a multi-layer diaphragm with a diaphragm-rupture warning system. The reproducibility of the metering is better than ±1% in the stroke length range of 20 to 100%. — *ProMinent GmbH, Heidelberg, Germany*

www.prominent.de

Save time and water with this tank-cleaning machine

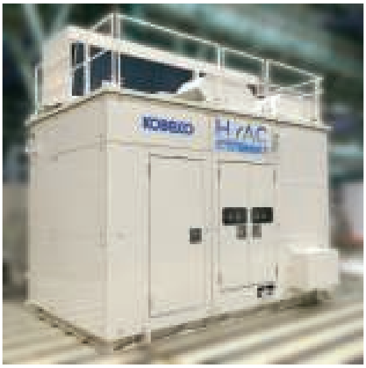
The new TJ40G rotary tank-cleaning machine (photo) uses a high-impact jet stream to effectively clean tough tank residues and minimize the risk of product contamination. This four-nozzle rotary jet head also cleans tanks 60% faster than static spray-ball technology, thereby increasing production uptime. And because it cleans faster, this new device uses less water and less cleaning agents which reduces operating costs by up to 70%, says the company. The TJ40G is capable of handling tough tank residues, as well as solids up to 1 mm in the cleaning fluid in tank sizes of 50–1,000 m^3. This is particularly important for demanding process lines, such as applications within a brewery, where particles may be re-circulated in cleaning media before completing the cleaning cycle. Also, the unit's hygienic self-cleaning construction ensures that the flow of the cleaning fluid reaches the exterior surfaces of the rotary jet head, as well as the critical interior components, including all bushings, bearings and inner surfaces. This minimizes the risk of product contamination and ensures high product quality.— *Alfa Laval AB, Lund, Sweden*

www.alfalaval.com

Predict product performance with a digital twin

The latest release of STAR-CCM+ software, v12.02, includes several new features to help product development organizations enhance and accelerate their ability to digitally simulate and understand how a product will perform in the real world using the digital twin — a precise virtual model of a product's physical and performance characteristics. New analysis tools combined with enhanced visual realism enable STAR-CCM+ v12.02 to help users unlock deeper meaning behind complex engineering simulations. STAR-CCM+ v12.02 introduces ray tracing capability that allows engineers to apply photo-realistic renderings to their design and simulation results. In addition to providing new tools for the analysis of simulation results, STAR-CCM+ v12.02 adds capabilities to accelerate simulation

Kobe Steel



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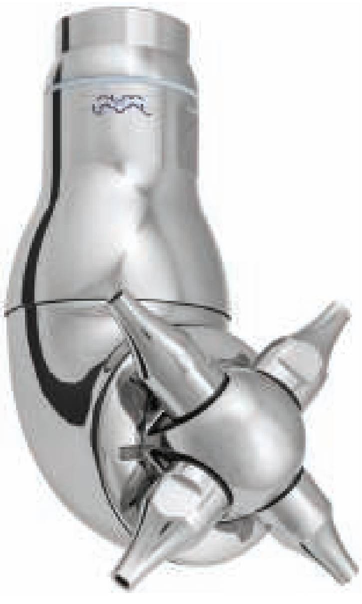

Alfa Laval

Kral

EM-Technik

Pittsburgh Corning

Charles Ross and Son

throughput for products that deal with reacting flows, such as furnaces, reformers, internal combustion engines and gas turbines. — *Siemens AG, Munich, Germany*
www.siemens.com

Flow measurement at low temperatures

Components and assemblies are often tested in cold chambers at temperatures down to –40°C. This company can equip its Volumeter flowmeter (photo) with electronic sensors that are suitable for such low temperatures upon request. The upper temperature limit is 200°C. For even measurements in extreme cold, the special advantages of the device's "screw" measuring method offers advantages over other methods, such as gear or Coriolis. In particular, the Volumeter offers a high measuring precision (±0.1% of the measured value) and a measuring range of 1:100. This wide measuring range makes it possible to pass through the broad characteristic curves during the functional testing of hydraulic components. — *Kral AG, Lustenau, Austria*
www.kral.at

Automated switch for multiple gas-analyzer lines

Up to now, solenoid valves have been used for switching between lines used for measuring gases, but a considerable effort is required for servicing such arrangements in the event of a failure. This company has developed an alternative — a new, automated measuring-point switch (photo). These point switches can be used in enclosed spaces, such as cleanrooms, and are located upstream of the gas analyzers. The switch can monitor 8, 10 or 16 measuring points. The device clocks all active sampling boxes with a pre-defined interval. In the event of an alarm, the measuring points can be switched manually or individually in order to determine the exact location of the gas emission. The automated measuring switch is equipped so that eight measuring-point changeover switches can be connected to each other. — *EM-Technik GmbH, Maxdorf, Germany*
www.em-technik.com

A new anti-abrasive coating for insulation

Pittcote 16 coating (photo) is a new low-temperature, anti-abrasive (LTAA) product that is designed for application with this company's Foamglas insulation blocks, curved segments (PSG), half shells (PSH) and fitting covers. Pittcote 16 LTAA coating is a water-based, rubberized latex resin that provides a durable bore coating to insulation substrates and reduces abrasion from vibrating piping or equipment. Due to its quick drying and brush/spray application capabilities, the product also reduces production time and increases efficiency. The coating can be used on a wide range of service temperatures, from cold and cryogenic equipment to above ambient (up to 120°C), allowing for reduced inventory requirements. — *Pittsburgh Corning, Pittsburgh, Pa.*
www.pghcorning.com

This closed-system mixer requires no transfer steps

The VersaMix Multi-Shaft Mixer (photo) is designed for mixing pastes, creams and gel-type products, with the capabilities of powder dispersion, emulsification, dissolution, deagglomeration, homogenization, heating, cooling and deaeration. All of these functions are achieved in a closed system, without the need for any transfer steps. The mixer's three-wing anchor, high-speed disperser and high-shear rotor-stator arrangement operate independently at different speed ranges to deliver uniformity in both low- and high-viscosity media. All product-contact surfaces are polished 316L stainless steel. The mixer cover includes multiple ports for charging raw materials and the installation of a vacuum or pressure transmitter, thermoprobe and tank light. It mates with a jacketed cone-bottom vessel that is designed for 100% discharge through a flush diaphragm valve. A 23-in. clearance from the valve outlet to the floor allows operators to position a bucket under the vessel to catch finished product after the mixing cycle. — *Charles Ross and Son Co., Hauppauge, N.Y.*
www.mixers.com ■

Mary Page Bailey and Gerald Ondrey

Facts At Your Fingertips

Gas Detection

Department Editor: Scott Jenkins

Detection of gases is a critical task in many chemical process industries (CPI) facilities to avoid hazards to personnel and to the environment. Gases may present risks of explosion, flammability, toxicity, environmental pollution and displacement of breathable air. This one-page reference provides information on common classes of gas detectors and on commonly monitored gases in industry.

Gas sensor classes

Gas-detection technologies can be classified according to the characteristics of the gases they detect: either toxic gases or combustible gases, and most gas-detection technologies fall into one of four broad categories based on their mode of operation: electrochemical sensors and metal-oxide semiconductor sensors are generally used to detect toxic gases; and infrared and catalytic sensors are used for detecting combustible and explosive gases.

Electrochemical. Electrochemical sensors are based on an electrochemical cell whose current increases when the molecule of interest makes contact with the sensing electrode. The target gas may be oxidized or reduced at the working electrode, and a small, but detectable flow of electrons is produced from the reaction there. A measuring electrode and a counter electrode are connected to the cell as well.

Metal-oxide semiconductor (MOS). Metal-oxide semiconductors are based on the principle that gas adsorption onto, and desorption from, the surface of a metal oxide changes the conductivity of the material. When target molecules contact a thin film of high-surface-area sensor material, the concentration of charge carriers (electrons or holes) changes, and the conductivity or resistivity is altered in a measurable way.

Catalytic. Most sensors of this type work by catalytic oxidation, where the combustible gas of interest comes into contact with a catalytic surface (often platinum-treated wire coil) and is oxidized. This releases heat of reaction, and the wiring resistance is changed by the temperature rise. Typically, a bridge circuit is used to indicate the resistance change. The increased resistance — compared to the resistance in clean air — is used to indicate the gas concentration.

Infrared. Infrared sensors work via a system of light transmitters and receivers. When combustible gases of interest come within the field of view of the receiver, a portion of the radiation is absorbed, changing the power of the light between the transmitter and the receiver.

Common detectable gases

The table [1] contains information on possible gas detection targets. ■

TABLE 1. COMMON GAS DETECTION TARGETS IN INDUSTRIAL SETTINGS

Gas name	Gas properties and notes	Exposure routes and common symptoms	IDLH[1] level; and REL[2]
Ammonia (NH_3)	Colorless gas with a pungent, suffocating odor. Often used in aqueous solution	Inhalation, (ingestion for the aqueous solution), skin and eye contact Irritation of eyes, nose and throat; difficulty breathing; pulmonary edema	300 ppm; TWA[3] 25 ppm
Carbon dioxide (CO_2)	Colorless, odorless gas. Shipped as compressed, liquefied gas. Solid form used as dry ice	Inhalation (for liquid and solid forms, skin and eye contact) Headache, dizziness, paresthesia, difficulty breathing	40,000 ppm; TWA 5,000 ppm
Carbon monoxide (CO)	Colorless, odorless gas. Shipped as compressed liquefied gas	Inhalation Headache, rapid breathing, weakness, exhaustion, dizziness, confusion	1,200 ppm; TWA 35 ppm
Chlorine (Cl_2)	Greenish-yellow gas with pungent, irritating odor	Inhalation; skin and eye contact Burning of eyes, nose and mouth, discharge of tears, cough, nausea	10 ppm; 0.5 ppm ceiling at any time
Chlorine dioxide (ClO_2)	Yellow to red gas (above 52˚F) or red-brown liquid with an unpleasant odor	Inhalation; skin and eye contact Irritation of eyes, nose, throat; cough, wheezing, bronchitis, pulmonary edema	5 ppm; TWA 0.1 ppm
Hydrogen chloride (HCl)	Colorless to slightly yellow gas with pungent, irritating odor. Often found as aqueous solution	Inhalation; skin and eye contact; ingestion for solution Irritation of nose, throat, larynx; cough, choking; dermatitis	50 ppm; 5 ppm ceiling at any time
Hydrogen cyanide (HCN)	Colorless or pale-blue liquid or gas (above 78°F) with a bitter, almond-like odor. [Note: Often used as a 96% solution in water.]	Inhalation, skin absorption, ingestion, skin and/or eye contact Asphyxia; weakness, exhaustion, headache, nausea, vomiting; increased rate and depth of respiration or respiration slow and gasping	50 ppm; Short-term only 4.7 ppm
Hydrogen sulfide (H_2S)	Colorless gas with strong odor of rotten eggs	Inhalation; skin and eye contact Irritation of eyes and respiratory system; apnea, coma, convulsions; conjunctivitis, eye pain, discharge of tears, abnormal visual intolerance to light, dizziness, headache, weakness, exhaustion	100 ppm; 10 ppm ceiling at any time
Phosphine (PH_3)	Colorless gas with a fish- or garlic-like odor	Inhalation Nausea, vomiting, abdominal pain, chest tightness, difficulty breathing, muscle pain, chills	50 ppm; TWA 0.3 ppm
Sulfur dioxide (SO_2)	Colorless gas with a characteristic, irritating, pungent odor	Inhalation and skin and eye contact Irritation eyes, nose, throat; rhinorrhea (discharge of thin nasal mucus); choking, cough; bronchoconstriction	100 ppm; TWA 2 ppm
Nitrogen dioxide (NO_2)	Yellowish-brown liquid or reddish-brown gas (above 70°F) with a pungent, acrid odor.	Inhalation, ingestion, skin and eye contact Irritation of eyes, nose, throat; cough, frothy sputum, decreased pulmonary function, chronic bronchitis, breathing difficulty; chest pain; pulmonary edema	20 ppm; 5 ppm ceiling at any time

[1] The term IDLH (immediately dangerous to life or health) is defined by the U.S. National Institute for Occupational Safety and Health (NIOSH) as exposure to airborne contaminants that is "likely to cause death or immediate or delayed permanent adverse health effects or prevent escape from such an environment."

[2] REL refers to recommended exposure limit, an occupational exposure limit that has been recommended by NIOSH and the Occupational Safety and Health Administration (OSHA) for adoption as a permissible exposure limit.

[3] TWA is the time-weighted average, used to indicate the average exposure over a specified time period (usually 8 h, unless otherwise indicated)

References

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Bio-based Adipic Acid Production from Glucose

By Intratec Solutions

Adipic acid is one of the most commercially important aliphatic dicarboxylic acids. It is produced on a large scale primarily to supply the nylon 6,6 production chain. Other applications include the manufacture of coatings, synthetic lubricants, fibers, plastics, plasticizers and polyurethane resins.

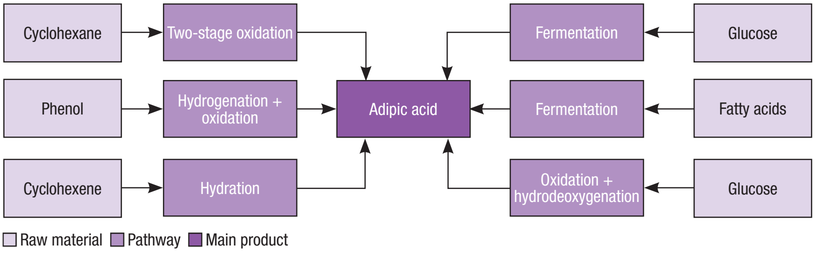

FIGURE 2. Multiple production pathways exist for adipic acid

The process

In the process described in the following paragraphs, adipic acid is produced from glucose via a two-step catalytic process (Figure 1).

Glucaric acid synthesis. A glucose solution is mixed with process water and heated before being fed to the oxidation reactor. Oxygen is supplied to the reaction by feeding compressed air to the bottom of the reactor. The overhead from the reactor is fed to a knock-out drum, a liquid stream is recycled to the reactor and most of the recovered gaseous material is recycled to the air compressor. The reactor's bottoms product is cooled and filtered, and glucaric acid is recovered as a solid. It is then fed to rotary dryers to remove water. Dry glucaric acid is conveyed to the hydrodeoxygenation feed mixer.

Adipic acid synthesis. Glucaric acid is dissolved in acetic acid and heated before being fed to the hydrodeoxygenation reactor. Glucaric acid reacts with hydrogen at high pressure, yielding adipic acid and water. The reactor's overhead stream, a hydrogen-rich stream, is partially condensed to remove light byproducts, which are burned as fuel. The gaseous stream is recycled to the hydrogen compressor. The reactor's product stream is expanded in a turbo generator, producing electricity. Its low-pressure output is directed to a knock-out drum in which a hydrogen-rich gaseous phase is recovered and recycled to the hydrogen compressor and a liquid phase, composed mostly of acetic acid, water and adipic acid, is directed to a crystallizer. The output from this crude crystallizer is then filtered and fed to a rotary dryer to recover crude adipic acid. The mother liquor from the filter and the acetic acid evaporated from the dryer are mixed and fed to an extractive distillation system to remove the water content from the acetic acid so it can be recycled to the hydrodeoxygenation feed mixer. Crude adipic acid must still be dissolved in water and then subjected to further crystallization, filtering and drying to reach fiber-grade purity.

Adipic acid production pathways

Adipic acid production has been predominantly based on cyclohexane and, to a lesser extent, phenol. Shifts in the hydrocarbon market and growing environmental concerns have resulted in the development of alternative production routes for adipic acid from renewable resources, such as sugar and fatty acids (Figure 2).

Economic performance

Using data from Q3 2013, variable costs (raw materials and utilities) for manufacturing fiber-grade adipic acid from glucose in the U.S. are estimated to be around $1,100/ton of product, accounting for a significant portion of the production costs.

This column is based on "Bio-based Adipic Acid Production from Glucose – Cost Analysis," a report published by Intratec. It can be found at: www.intratec.us/analysis/adipic-acid-production-cost. ■

Edited by Scott Jenkins

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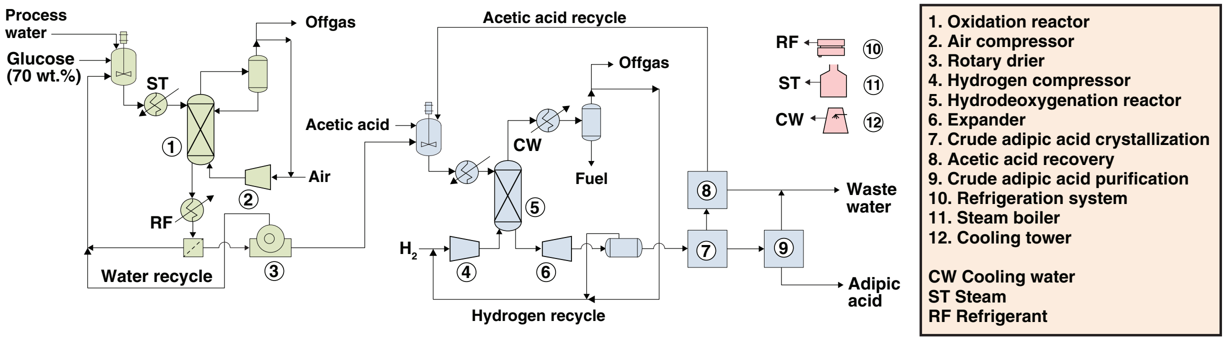

FIGURE 1. The production of bio-based adipic acid from glucose via a two-step catalytic process is shown here

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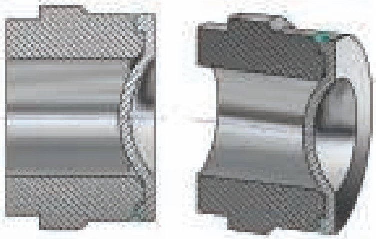
Under Pressure - how to safeguard high pressure processes



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Part 1

Batch Drying With Vacuum Contact Dryers

Drying is a demanding unit operation where solvents need to be removed without altering product chemistry. Vacuum dryers are essential pieces of equipment for accomplishing this task

Eberhard Tritschler
Ekato Systems GmbH

IN BRIEF

Drying solids is a demanding task that requires both knowledge of process technology and a sound mechanical design of the equipment used. This article provides an overview of batch-drying principles and possible options for vertical vacuum-contact dryers.

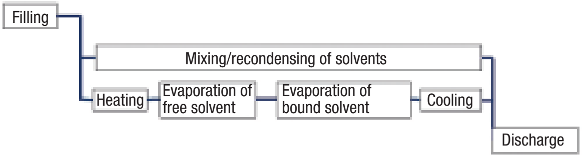


FIGURE 1. Batch drying processes typically consist of the components and steps shown here

Most solids in the chemical process industries (CPI) are recovered by precipitation or crystallization. The suspended solids are mechanically separated by filtering or centrifuging in the first instance. In most solid-liquid-separation processes, thermal evaporation does not compare favorably with mechanical separation by filtration or centrifugation with respect to energy efficiency. However, when it comes to the final moisture content, thermal evaporation is often the follow-up step to mechanical dewatering methods in order to achieve the desired dryness.

The residual moisture content of the solids after filtration or centrifugation is influenced by the properties of the solid's crystal structure. A typical filtration cake can show residual moisture content of between 5 and 45%. To dry a wet cake to lower values, thermal evaporation is an effective way to achieve a true "dry" solids powder.

For some products, continuous drying processes, such as belt and conveyor drying or spray drying, are suitable methods to achieve a dry powder in a single pass. While continuous systems are not a subject of this article, more information can be found in Part 2 of this Feature Report, "Optimizing Analysis for Spray Drying Processes" (p. 43).

Vacuum drying

During any drying process, all additives and solvents have to be removed from the solid material without changing its original chemical composition. In essence, there must be no changes to the molecular structure during the drying process and no derivatives formed due to the drying process.

A vacuum contact dryer is built to evaporate solvents from a solid by the use of the following physical and mechanical parameters: temperature; low pressure; and mechanical fluidization by agitation.

In most cases, vacuum contact dryers are restricted by the requirement that they not influence the chemistry of the product. However, special processes are possible, where the application of high temperature will initiate or control a chemical reaction.

The degree of residual moisture in the finished product is strictly related to the use of the product and its specifications. Examples include the following: less than 5% moisture for waste material for disposal; less than 3% in foodstuffs for storage considerations; less than 1% in herbal extracts; and less than 0.1% in active pharmaceutical ingredients (APIs).

A vacuum contact dryer can achieve these values for the separation of solids from solvents and there are cases where the solids, the solvents or both, are valuable products.

Batch drying process

Almost all batch drying processes consist of the process steps mentioned in Figure 1. Whether the steps are performed in a non-agitated tray or in an agitated vessel, the concepts discussed here for evaporation and re-condensing and collecting the solvents are the same.

Each process step in batch drying has individual issues that require special attention:

Filling. During filling, factors such as the use of gravity or force (pumping), as well as the need for a contamination-free transfer into the dryer will have to be addressed.

Mixing. In order to create an even temperature and solvent-concentration distribution in the bulk material, as well as to support the outflow, mixing is mandatory in the dryer. However, the influence of shear forces created by the mixing action on the solids needs to be addressed.

Re-condensing. Whether the purpose of the drying is to retain the dry power and discard the solvent, or vice-versa, is determined by the user. Re-condensing and collection of the solvents is a demanding task in the process.

Evaporation of free solvent. The solvent selection dictates the evaporation conditions (enthalpy) and the maximum temperature limits of the solids dictates the thermal limitations for the drying system.

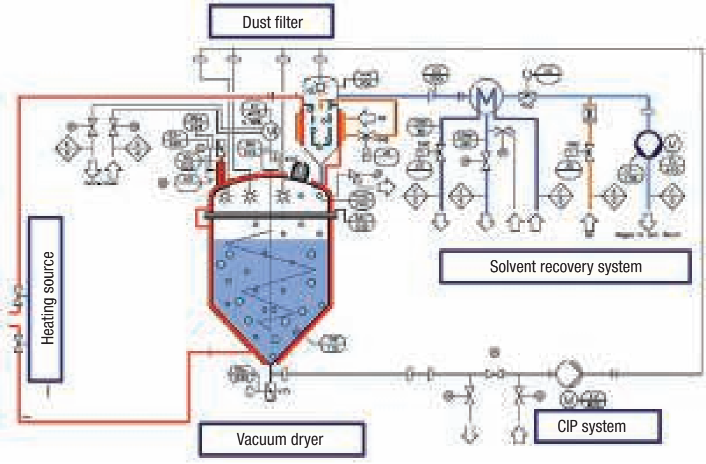


FIGURE 2. Vacuum drying systems require several pieces of key equipment

Evaporation of bound solvent. Some solvents tend to stick to the solid particles by physical bridging, or to hide in the pores of the particle structure, while others don't. In order to predict required process times, detailed knowledge of the separation behavior is critical.

Cooling. For safe discharge, it might be required to cool the solids to very low tem-

TABLE 1. EXAMPLES FOR IDENTIFYING PROCESS LIMITATIONS

Product	Process	Solvent	Vacuum level	Max. temperature
Polymers	Post treatment	Water	Med. (50 mbar abs)	High (300°C)
Pigments/flame retardants	Vacuum drying	Water, organics	Med. (50 mbar abs)	Average (80–100°C)
API	Post drying	Water, organics	Low (8 mbar abs)	Low (40°C)

peratures prior to discharge. Since the heat removal from a dry power is subject to even lower heat-transfer coefficients, the cooling step might be time consuming.

Discharge. While fluids or suspensions drain well from a vessel, draining a dry powder can be a challenge. Residues after draining are lost in most instances and reduce the cost efficiency of the dryer.

In addition to the aforementioned basic requirements, supplemental codes and standards, such local laws, pressure vessel regulations, good manufacturing practice (GMP) rules, pharmaceutical standards, U.S. Occupational Safety and Health Administration (OSHA) requirements and explosion protection measures have to be considered.

Equipment overview

The following section is intended to identify the key components of a vacuum drying system (Figure 2).

Heat source. With respect to the heating source for the dryer, available site utilities, as well as product specifications have to be considered. While a supplier of aluminum pigments might ban water as a heating fluid for fire hazard reasons, an API producer might avoid thermal oil to avoid contamination risks. Both are secondary aspects, not directly related to the physical operation of heating.

The base specification for a dryer heating system should consider the following:

- Provide a sufficient amount of heat (Joules) in an energy efficient way
- Provide measures to keep a constant temperature in the system

The correct heating power and capacity calculation has to consider the following:

- Initial heating of all wetted components (equipment) within a reasonable amount of time
- Provide sufficient energy to heat the product load to the operating temperature
- Provide sufficient energy to evaporate the solvent load at the given conditions
- Compensate for energy losses
- Maintain temperature

In most cases, circulating a clear fluid in a closed loop is the appropriate method for transporting the energy from the heat source to the dryer vessel. For thermal fluids, water, water-glycol mixes or thermal oils are common.

For its high specific-heat capacity, water is the first choice as a thermal fluid when the temperature needs to stay below 150°C. At process temperatures above 150°C, thermal oil becomes more feasible, since oil shows only a marginal pressure rise at elevated temperatures.

Whether the closed loop is driven by one heat exchanger for heating and one heat exchanger for cooling, or the system is fed by a centralized plant heating and cooling system is to be decided based on the site conditions. Proper sizing is important.

Prior to any equipment selection, the physical limitations for the process need to be defined by the process owner. Each and every process has special demands, including (but not limited to) the three examples in Table 1.

Vacuum dryer design

The majority of commercially available vacuum contact dryers are built according to the same principle: a jacketed vessel — either horizontal or vertical — with a wall-wiping agitator. Vertically oriented dryers can take advantage of gravity to influence product flow (Figure 3). Vertical vacuum-contact dryers include the following:

- An upright cylindrical vessel with a filling-to-height diameter ratio designed for bulk mixing
- A cone bottom to support the outflow of solids
- A jacketed to suit the selected heating system
- A top-entry agitator, designed for solids applications (re-starting in settled solids)
- Valves and safety applications to operate a pressure vessel

Solids impellers circulate the entire contents of the vessel from the gravity filling until the gravity discharge. The impellers can be hydrofoil type, designed to create maximum flow with the lowest possible power input.

Energy balance is important. Mixing power by the impeller equals the power into the process. In a temperature-sensitive process, excessive mixing power input might disturb the fragile temperature balance in the product.

The key feature of all agitators in a vacuum contact dryer is an agitator system that operates with minimum wall clearances and wipes the full wetted surface of the vessel. Wall clearances from 5 to 15 mm are common and feasible, and require both a precisely fabricated vessel and a deflection-free balanced impeller.

Since the process is typically operated under vacuum, the vessel must be fabricated according to the American Society of Mechanical Engineers (ASME) Boiler and Pressure Vessel Code (BPVC), and must also follow local laws and regulations, as applicable.

Dust filter

By heating a wet solid in the closed vessel, evaporation will start. The solvents will be condensed in an external solvent-recovery system. This migration of solvents will create a strong gas flow that may cause dust formation as the solids dry.

Commercially available solvent-recovery systems are built for heavy duty services, but not for high amounts of dust. In order to prevent the plugging of lines — or simply to keep the solids where they belong, in the dryer vessel — a dust filter is installed on top of the dryer vessel.

Modern dryers feature heated filter domes with automatic blowback cleaning systems that are properly designed to protect the solvent-recovery system from the dust.

Two types of commercially available filter elements are discussed here, but others' experience might include many more systems.

The dust-loaded area of the dryer is separated from the solvent recovery line by a gas-tight filter plate. This filter plate carries the filter elements. One possibility is a fabric bag filter mounted on a wire cage, while the other is a sintered metal or wire mesh (10 µm) cartridge made of metal wire or metal fibers

The selection of one of these filter systems is based on owner preference and on the overall operating conditions (including temperature). While the fabric bag filters are disposable and low in replacement cost, the metal cartridge is reuseable with a high lifetime expectation.

In both cases, proper sizing of the filter is critical. An undersized filter will lead to an excess pressure drop in the solvent-recovery line, with effects on the total evaporation regime.

FIGURE 3. Vertical solids dryers can take advantage of gravitational forces for solids flow

TABLE 2. TYPICAL EVAPORATION TEMPERATURES FOR WATER

Water	Pressure, mbar abs	Boiling temperature, °C
	1,013	100
	100	45
	10	5

TABLE 3. TYPICAL EVAPORATION TEMPERATURES FOR ETHANOL

Ethanol	Pressure, mbar abs	Boiling temperature, °C
	1,013	82
	100	32
	10	5

TABLE 4. TYPICAL DRYING BEHAVIORS

System	Step 1	Step 2	Step 3
Pigment drying	Evaporate surface moisture	Transition	Evaporate crystal moisture bound in the capillary
Comment	High solvent load, low dust emission	Dust emission starts	Long duration, low solvent load, dust formation

Solvent-recovery system

As the vacuum contact dryer evaporates the solvents, the solvent-recovery system needs to re-condense the solvent, usually at the same rate. There are many ways to achieve this objective. This article covers one method that has shown some advantages when it comes to collecting the solvent for further use (Figure 4).

A robust vacuum pump (liquid-ring pump or dry-running pump) creates the initial low pressure that will activate the process. Design criteria for the pump includes the following:

- Evacuate the total system volume in a reasonable time
- Maintain the vacuum level under changing process conditions
- Compensate for air leakage across the full system
- Compensate for inert gas volumes introduced during the process

In essence, the vacuum pump has to keep the system running while also covering for losses.

The key component of the solvent-recovery system is the heat exchanger or condenser that is installed on the low-vacuum side of the pump. Properly designed and activated with the proper cooling fluid, solvents evaporated in the hot dryer vessel will migrate to the cold surface of the heat exchanger and will be re-condensed.

Condensation on the vacuum side of the pump will result in a huge draft — the condenser acts as a "vacuum pump." The liquefied solvents are collected in a collection vessel for further use or transfer. Cooling or insulating the vessel is recommended when the system is installed at higher ambient temperatures.

FIGURE 4. The solvent-recovery system recondenses the solvents as the dryer evaporates them

With respect to the cooling methods, again the circulation of a clear fluid is one of the first choices. Based on commonly used solvents and the vacuum level achieved, anything from water (at 8°C) to brine (at –15°C) might be required. One item needs to be considered: water at 25°C should not be considered "cold."

Collecting the solvents is the most precise method for back-calculating the residual moisture in the dry product. This can be done simply by measuring the solvent volume. A small amount of solvents, however, might escape even a properly designed condenser. To catch the saturated solvents, a second condenser in the exhaust line of the vacuum pump might be required.

Process controls

All of the components mentioned in this article require safe and precise operation. The large number of signals to be processed and valves to be handled does not allow manual operation of such a system, except situations involving small-scale tests in a laboratory environment. Automatic process control systems that focus on a proper temperature and vacuum regime are mandatory for industrial vacuum drying.

Drying process steps

As noted earlier in the article, an effective drying process requires knowledge of the full picture of the process, including filling, mixing, re-condensing, evaporation of free solvent, evaporation of bound solvent, cooling and discharge of products

Filling. There are many ways of transferring a solvent-loaded wet cake into a dryer vessel. This could be the subject of an article on its own, covering all aspects such as contamination, operator exposure levels, explosion risks and dosing the correct amount. Some dryers provide a wide opening at the top of the dryer vessel where valves, dosing or conveying systems, including (but not limited to) material-containment apparatus, can be installed. Gravity will support the filling process.

Mixing. During the filling process, the mixer may or may not run. However, it has to be designed for re-starting in settled solids at any time during the process. Stalling during a drying process should be avoided. In some rare cases, however, stopping the agitator is even required to overcome a certain process condition. In essence, an "off-the-shelf" agitator will not do the job. Dryer agi-

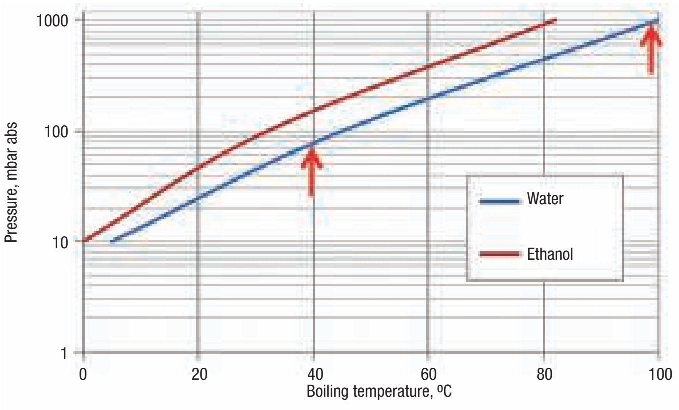


FIGURE 5. Solvent evaporation behaviors can be plotted in graphs like the one shown here

tators have to be designed for the purpose.

The degree of mixing might or might not affect the drying time. It is the task of the scale-up engineer to predict the influence of the mixing on the drying behavior. Engineers must gain this information in a small-scale test.

Re-condensing. Evaporation starts when the solvent vapor pressure exceeds the pressure in the vessel or the product temperature exceeds the boiling temperature of the solvent.

Since the heat transfer into a mixed solids bed is difficult (because of low overall heat-transfer coefficients of about 50 W/m²K), additional measures are required to increase the system efficiency. Often the dry product shows temperature sensitivity, limiting the allowable heating fluid temperature.

In order to compensate for both, the limitations in heat transfer and heating temperature, working under vacuum is advised. By lowering the pressure in the vessel, the boiling temperature of solvents drops. Typical evaporation temperatures for water and ethanol are shown in Tables 2 and 3.

Water has a vapor pressure of 1,013 mbar abs (~760 torr) at 100°C, so its vapor pressure would exceed atmospheric pressure at temperatures higher than 100°C and the water starts boiling (Figure 5).

If a desired solid product is water-loaded, but melts above 40°C, it is advisable to ensure that the heating-system temperature does not exceed 40°C. The vapor pressure of water at 39°C is 70 mbar abs. To avoid melting during the drying process, the pressure inside the vessel has to be set to less than 70 mbar abs.

Drying curve. Once the theoretical parameters have been set, a small-scale test is recommended to gain sufficient information about the exact drying behavior of the product. The following are characteristics for typical small-scale testing of drying behavior:

- 3-L scale: Yes/No-type testing with limited data collection for scaleup
- 25-L scale: best for free-flowing products; results allow safe scaleup to plant size
- 50–100-L test: best for difficult-flowing products; results allow safe scaleup to production scale

During a drying test, in the first instance, proof that the agitator is suitable to move the product is mandatory. All process-related parameters must be collected in order to generate the drying chart, a plot that shows the progression of the solvent content over the drying time (Figure 6).

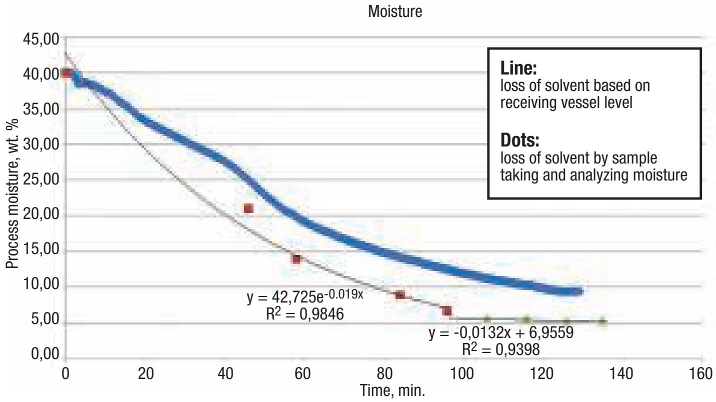
For this example process, the solvent evaporates at an almost constant pace until reaching the goal of 5% solvent by weight. Based on the results of this chart, the following parameters affecting the hardware are calculated:

- Required power consumption of the heating system (amount of evaporation heat in relation to the time required)
- Required dust filter sizing, with respect to the solvent load over the evaporation time
- Required vacuum condenser sizing, with respect to the solvent load over the evaporation time

Each and every drying chart looks different for the great number of drying applications. However, they all give an indication about typical drying behaviors and are divided into three sections (Table 4).

Discharge. When the process temperature is equal to the heating temperature, this is an indication that energy is no longer being spent for evaporation, and the drying process can stop. However, safety considerations might not allow the discharge of a hot solid. In many cases, cooling prior to discharge to lower temperatures is mandatory. Some dryer systems have the ability to change the heating system to a cooling mode. While the heat transfer for heating already has very low values, the values for cooling a dry powder are even lower. Cool-

FIGURE 6. Drying curves show the progression of solvent content in the solid material over the drying time



ing the dry solids will consume additional production time. In addition, the stainless-steel mass of the dryer equipment also needs to be cooled. In cases where time is of the essence, it might be beneficial to separate the functions and transfer the hot solids by closed pipe into a designated cooling vessel directly below the dryer. This procedure saves the energy required to cool the equipment mass.

Maximum yield after discharge is important for the cost balance of the drying system. In a single-product manufacturing process, leaving residues in the drying vessel for the next batch may be permitted. However, in a typical pharmaceutical or fine chemicals process, "cross-contamination" of products is banned.

With a cleverly designed vessel and a flexible agitator, the equipment can be made to suit the flow characteristics of the product. A vertical vessel will, by nature, drain better than a horizontal drying vessel. A cone bottom supports outflow far better than a flat or dished bottom. The agitator must be in a position to rotate at elevated speeds, including the changing the direction of rotation for discharge. Also, surface polishing and surface linings can help to reduce friction as the solid product flows.

Scaleup. Scaling up a drying process requires both small-scale testing capabilities and scaleup experience. Once successfully dried at a 25-, 50- or 100-L scale, scaleup rules can be used to predict the following:

- Drying time
- Solvent load, energy demand
- Residues after discharge
- Particle size changes due to shear on the product
- Wear of the agitator parts due to exposure to the product

As a basic rule, a safe scaleup requires that the geometry of the small-scale test dryer is geometrically similar to the production-size dryer.

Scaleup works best when the trials have been carried out under identical physical parameters (such as pressure and temperature) as the full-scale process. If this is not possible, correction factors have to be applied to the scaleup formula. The scaleup rules consider the following items:

- The ratio of volume-to-heated-surface-area of the small-scale dryer to the full-scale dryer
- A temperature correction factor in moving from small scale to full scale, if the values deviate from one another
- A factor to account for the influence of agitation speed on the evaporation

If all data are available, safe scaleup by a factor of 200 is readily achievable.

Concluding remarks

The information included here describes the basic concept of a vacuum contact-drying system. This type of system can be applied to many chemical applications by designing the components accordingly. The following are some of the typical situations:

- Selection of wetted parts with respect to corrosion (most commercially available metals)
- Designing the components for GMP standards (food and pharmaceutical industries)
- Designing the components for critical solvents, such as organic solvents
- Designing the components for severe site conditions (outdoor and weather protected)
- Designing the components for high demands on cleanability (cleanroom environment)
- Designing liquid cleaning systems (clean-in-place; CIP) for the dryer vessel
- Designing the components for out-of-specification applications (high temperatures and pressures)

Aside from plant experience and knowledge of the process, cooperation with a capable dryer company opens a wide range of possibilities beyond vacuum contact drying. Applications include solvent extraction from herbs and spices; all-in-one reaction systems for paints and pigments; food pasteurization and drying; and all-in-one chemical reactor/dryers. ■

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Part 2

Optimizing Analysis for Spray-Drying

Precise control over spray-drying processes can be enabled by modern measurement approaches that provide information on particle size and shape in realtime

FIGURE 1. Atomization technolgy is critical for spray drying processes. These photos show rotary atomizers (left), two-fluid nozzles (middle) and pressure nozzles (right)

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The process of spray drying is extremely valuable to a wide range of solid-product manufacturers due to its ability to produce particles with precisely controlled size and shape. Cutting-edge spray drying technology is used in industries ranging from food and pharmaceuticals, to industrial materials, metal powders and chemicals. Spray drying technology is typically selected for its ability to deliver standardized particles, so optimizing analytical strategies for process monitoring is crucial to its successful implementation. Methods for monitoring spray drying have developed significantly in recent years, with new technologies making it easier to achieve precise control. In this article, we look at those techniques that are proving particularly valuable, focusing on online particle sizing and automated imaging, a technology that quantifies particle shape, as well as size.

Spray drying advantages

Spray drying produces a dry powder from a liquid solution or slurry. The liquid is sprayed into a drying chamber in which air, or an inert gas, drives off the solvent to leave a dried powder; subsequent classification ensures a closely defined particle size distribution. A spray drying process can be operated as either an open system, in which filtered air exits to the atmosphere, or in a closed loop under inert gas, with solvent recovery, depending on the characteristics of the powder being produced and of the original slurry or solution.

Spray drying offers a number of significant advantages relative to other powder-production methods that give it broad commercial appeal. First, it produces highly spherical particles that consequently tend to have excellent fluidity. It also delivers particles or granules with a sharp, stable and closely defined particle-size distribution, offering a highly consistent output. Furthermore, spray-drying processes can be operated continuously, minimizing the manual input and potential for variability associated with batch processing (For more on batch drying, see part 1, "Batch Drying with Vacuum Contact Dryers," p. 36). These benefits bring value in the manufacture of products as diverse as milk powder, vitamins, antibiotics, cosmetics, pigments, ceramics and metal powders — including steel and rare-earth-metal-based products.

IN BRIEF

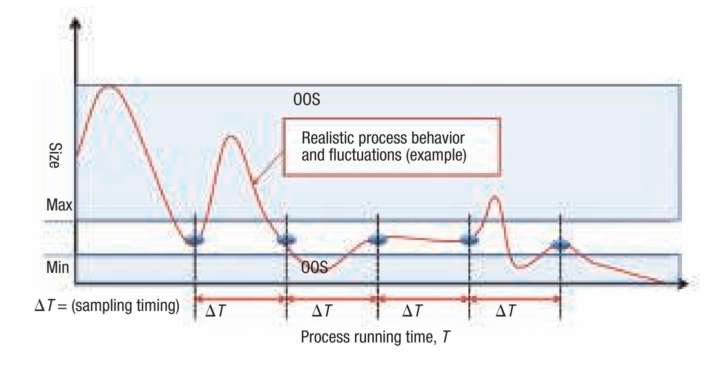


FIGURE 2. This graph illustrates the benefits of improved control over spray drying processes, including reduction of out-of-specification (OOS) material

Atomization technology

In order to tailor the properties of spray-dried materials to a specific application, it is important to select the most appropriate atomization technology, and then to optimize the process parameters that are available to exert control. Understanding the different commercially available atomizing equipment, and their method of operation, is a useful first step (Figure 1).

Rotary atomizer. A rotary atomizer applies the shear needed to break up the liquid solution into droplets via a rotating disc. Droplet size is controlled by the size and shape of the disc and, once the mechanical design is fixed, by varying the speed of rotation. This technology is most suitable for producing particles ranging from 20 to 200 µm in size, with the resulting products having high sphericity and narrow particle-size distributions compared to products generated with other spray-drying equipment.

Two-fluid nozzle. An alternative option for atomization within a spray-drying process is a two-fluid nozzle. With this technology, the energy required for liquid breakup comes from a flow of compressed gas, which is used to drive two-phase flow through the nozzle. Droplet size is controlled by manipulating the compressed gas pressure (flowrate). An advantage of two-fluid nozzles is that they produce very fine particles, from around 1 to 20 µm, and offer a process with a small physical footprint. However, a large quantity of compressed gas is required for operation.

Pressure nozzle. A pressure nozzle is another atomization technology used routinely in spray drying. A pressure nozzle atomizes the liquid simply by forcing it through an orifice under high pressure. This technology offers low operating costs and, via orifice selection, allows substantial scope to vary the size of the granules produced. Pressure plays an important role in defining the atomization process, thereby directly impacting the size of the droplets and particles produced. Reducing pressure increases the median particle size of the finished powder, while increasing it has the opposite effect.

Once the most suitable process technology has been selected, optimization of the spray-drying process relies on the effective manipulation of processing parameters and of the properties of the feed solution or slurry.

Important feed characteristics include the following: concentration, viscosity, specific gravity, additive content and inclusion, and primary particle-size distribution. All of these characteristics help determine the morphology of the finished particles.

Potentially influential process parameters include the following: the inlet and outlet temperature of the dryer, the feedrate of the liquid, and the air flowrate through the dryer and nozzle, depending on the atomization technology used.

Timely and informative particle characterization information drives the comprehensive optimization of these interacting process parameters.

Particle size and shape

The primary metric used to specify the properties of spray-dried particles is size, since this has a defining influence on performance and typically forms the basis of a product specification. The morphology of a particle is directly linked to the fluidity of the associated powder, a prized characteristic of spray dried products. Generally speaking, smaller particles flow less easily than those that are larger because interparticle forces of attraction increase with decreasing particle size. However, particle shape can also have a marked impact. Irregular-shaped particles can inhibit flow as a result of inter-particle friction or interlocking; particles with a regular shape more easily slip past one another, thereby flowing more freely. When it comes to controlling spray-drying processes, particle size measurement, though vital, is not all that is required. Particle shape data can also be extremely valuable.

Laser diffraction

Laser diffraction is used as a method for measuring particle size, and is usually the preferred option in the majority of spray-drying applications because it offers a number of important advantages over alternative methods. Laser diffraction is an ensemble particle-sizing technique, generating volume-based results for the whole sample rather than building up a size distribution from measurements of individual particles. As a result, it is inherently fast. Laboratory-based measurements take less than one minute.

Laser diffraction generates particle-size distribution data from measurements of angular variations in the intensity of light scattered by a dispersed sample as it passes through a laser beam. Large particles scatter intensely at relatively narrow angles to the incident beam, whereas smaller particles produce a lower intensity signal but at much wider angles. Laser diffraction analyzers record the angular dependence of the intensity of scattered light using a detector array, and from it calculate the particle-size distribution of the sample using an appropriate theory of light behavior — typically the Mie theory.

The technique has a measurement range of approximately 0.01 µm to 3.5 mm, comfortably covering the size range of interest for many spray-drying applications. Laser diffraction can report over 100 class sizes, delivering excellent resolution for the precise detection of variations in particle-size distributions. This technique can be automated to reduce requirements for manual input to a minimum, and to keep maintenance requirements for laboratory systems to a minimum, while simultaneously increasing repeatability and reproducibility.

Continuous particle sizing

The inherent speed of laser diffraction and its amenability to automation have led to the technique's successful commercialization as in- or on-line technology for continuous realtime particle sizing. Alternative realtime sizing techniques include spatial particle velocimetry, which determines the size of particles present from measurements of the duration for which they interrupt a laser beam. As already established, particle size is of crucial importance in the spray drying process, so the availability of robust systems for realtime monitoring can substantially enhance options for process optimization.

An on-line laser diffraction analyzer system can be easily integrated into the process and will then automatically measure particle size, providing a continuous stream of analytical data with no manual intervention. Eliminating the operator-to-operator variability associated with manual analysis and sampling improves repeatability and reproducibility, and the rate at which on-line systems analyze data — typically on the order of four complete particle-size distributions per second — is sufficiently fast to track even rapidly changing processes.

Because laser diffraction technology can be applied from "lab to line," specifications developed in a laboratory can be easily transferred into the manufacturing environment. Once a particle size specification is in place, realtime measurement helps to improve process control, making it easier to ensure that the process consistently produces particles of the required quality. Figure 2 shows how the data made available by continuous realtime measurement allow operators to robustly track process fluctuations and instantly see when the product is drifting out of specification (OOS). As a result, they can adjust the process to rectify a problem. This level of insight is not accessible to operators applying manual analysis, an approach that can easily result in OOS material being included in the final product. Furthermore, as well as improving manual control, automated measurement provides an essential platform for fully automated closed-loop control, opening up a route to further efficiency gains.

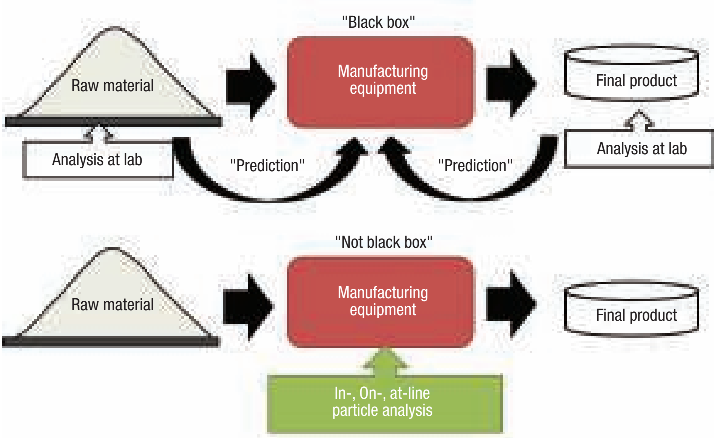
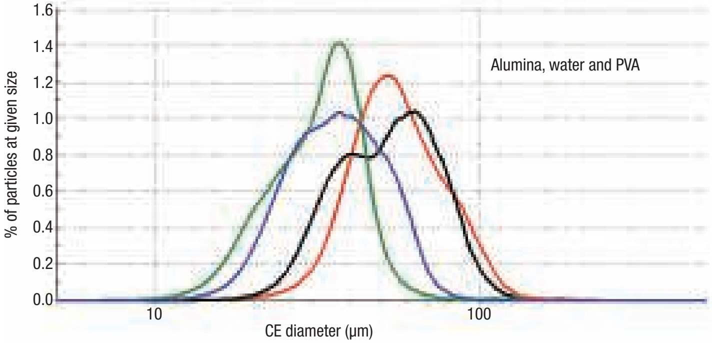


FIGURE 3. Realtime, continuous monitoring enables the assessment of quality of spray-dried products

Figure 3 summarizes the fundamental shift in process operation that realtime measurement delivers. In simple terms, continuous measurement makes it possible to assess the quality of spray-dried particles as they are being produced, rather than post-manufacture, prompting responsive, remedial

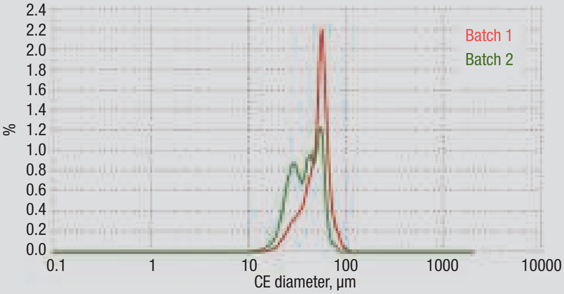
FIGURE 4. This process optimization test used a rotary atomizer for the spray drying process and shows the effect of speed on resulting particle size. The data suggest that a speed of between 6,000 or 8,000 rpm is sufficient to achieve the desired particle size of less than 50 µm



Blue, 12,000 rpm, Dv50 = 36 um: Green; 1,000 rpm, Dv50=32 um
Black; 8,000 rpm, Dv50 = 53 um: Red; 6,000 rpm, Dv50=56 um

THE IMPACT OF SOLUTION VISCOSITY ON PARTICLE SIZE

The amount of shear applied to the liquid feed during atomization in a spray-drying process is set by the design of the atomization technology and by the process parameters, such as pressure. However, the effect of that applied shear depends on the viscosity of the liquid. With higher-viscosity solutions, the shear has less impact than with a lower-viscosity feed. This means that, in the absence of any other change, an increase in viscosity will result in an increase in droplet size. An alternative way of looking at this is that solutions of higher viscosity require greater energy input to achieve successful atomization for an equivalent droplet size. The example discussed here shows clearly how a viscosity change can impact droplet formation and, consequently, can affect the morphology of the resulting powder particles.



Observing the impact of viscosity on the properties of spray dried particles. Because spray-dried materials are prized for their precisely defined properties, reproducibility is critical to the value of products manufactured. The graph here shows data for two batches produced under identical spray-drying conditions. The particle size Dv50 for the resulting granules for the two batches are 52 and 39 μm, respectively.

TABLE 1. MATERIAL DIFFERENCES AND SLURRY VISCOSITY

Batch	Dv50, μm	Slurry viscosity, mPas
1	50	2,200
2	39	350

Differences in the material have a significant effect on the slurry viscosity, and the size of the product

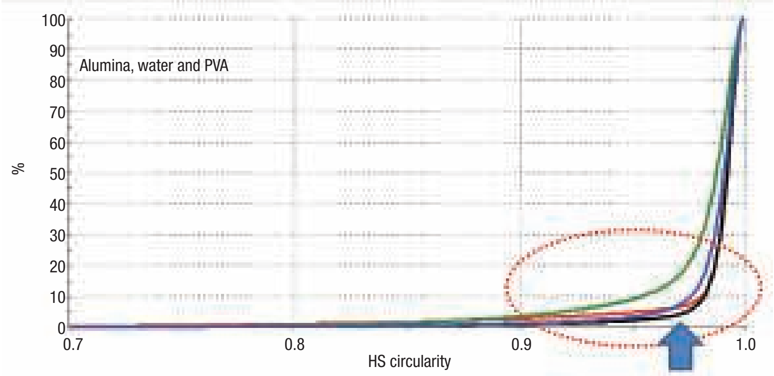
Investigations into the reason for the observed difference in performance highlighted a marked difference in the viscosity of the slurry used to produce each batch (Table 1). Though the stock solutions for both batches were produced by milling for identical periods of time using identically sized balls within the mill, the ball-mill media material and weight were changed between the two runs. This led to a substantial difference in the viscosity of the two stock solutions. The observed change in particle size is attributed to this difference. Establishing control over the ball-mill charge conditions corrected this issue and delivered the consistent viscosity required to ensure reproducible manufacture.

action to address an issue. Realtime analysis directly enables better process control — either automated or manual — thereby increasing throughput, reliability and reproducibility, while at the same time saving time and money by eliminating any requirement for manual measurements and control.

Including particle shape

As mentioned previously, beyond size, it is shape that defines the value of spray-dried products, and that is consequently a crucial consideration when selecting instrumentation for analysis. Automated imaging captures microscope-quality images of tens of thousands of individual particles in minutes and is a powerful technique for particle characterization. The resulting images are used to calculate number-based particle size and shape distributions, providing detailed, statistically relevant morphological analysis. Automated imaging offers the major advantage of allowing the user to view the particles produced and, compared with manual microscopy, is faster and far less subjective. The measurement range varies from approximately 1 μm to 10 mm.

When it comes to optimizing spray-drying processes, automated imaging is highly complementary to laser diffraction. It enables shape to be optimized along with size, for example, to access premium performance. Automated imaging can also be used for troubleshooting. The ability to quantify shape allows for the more robust differentiation of particles and can elucidate differences in performance between similarly sized products. Furthermore, the calculations that underpin laser diffraction analysis assume that the measured particles are spherical, so results can be affected by changes in particle shape. Automated imaging makes it straightforward to confirm that samples are adequately dispersed prior to laser diffraction analysis so that primary particles, rather than



Blue, 12,000 rpm, Dv50 = 36 um: Green; 1,000 rpm, Dv50=32 um
Black; 8,000 rpm, Dv50 = 53 um: Red; 6,000 rpm, Dv50=56 um

FIGURE 5. Shape data show that although 6,000 and 8,000 rpm are both sufficient for the production of appropriately sized particle, the faster speed ensures more spherical particles

agglomerates, are measured, and detects changes in shape that may skew the particle size data generated.

The following real-world examples illustrate how laser diffraction and imaging in combination support the optimization of spray-drying processes.

Case 1: Optimizing process conditions with respect to particle size and shape. Rotary atomizer technology was selected for the production of alumina particles and tests were then carried out to determine the optimum speed of rotation of the disk. The feed solution for the process was a mixture of alumina particles, water and polyvinyl alcohol (PVA), which was added as a binder within the suspension. In an initial test series, rotary speed was varied in the range 6,000 to 12,000 rpm and the size of the resulting particles was compared to the target specification, which defined the required Dv50 as being greater than 50 µm. From these results, a speed of rotation of either 6,000 or 8,000 rpm was identified as being suitable (Figure 4). A rotational speed of 6,000 rpm is preferable from the perspective of energy consumption. However, shape analysis reveals that in fact, these two speeds are not equally suitable in terms of the particle morphology of the resulting particles.

Shape data show that the higher speed of rotation (8,000 rpm, rather than 6,000 rpm) produces more spherical particles that, because of their shape, are likely to have superior fluidity (Figure 5). Here then, shape data provide additional, valuable insight into the liquid breakup process and its result, supporting process optimization to a more suitable conclusion.

Case 2: Studying process dynamics to define production conditions. A further study of the impact of disk-rotation speed on the size of spray-dried particles produced using a similar alumina-water-PVA suspension to the one tested in Case 1 was carried out using realtime size-monitoring technology, an on-line laser diffraction particle size analyzer. Here, tests at different speeds of rotation were carried out over a relatively short timescale, simply by stepping through the operating conditions of interest to determine those required to produce the target Dv50 (median particle size) and the time taken for the process to stabilize. The target Dv50 in this case was 55–65 µm, but the level of fines in the product was also a concern. The ability of the on-line system to accurately measure down to around 0.1 µm was therefore an important benefit.

Figure 6 shows that as the speed of rotation of the disk is stepped down, the particle

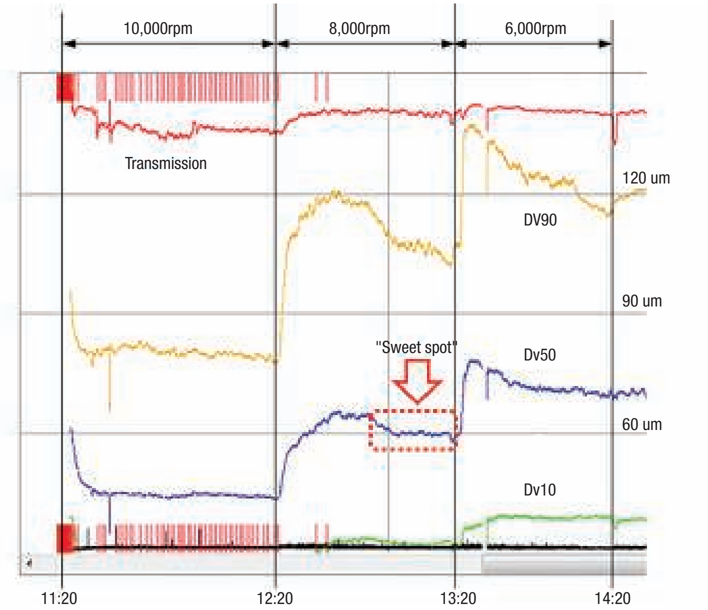


FIGURE 6. Realtime measurement makes it possible to instantly observe the effect of decreasing the speed of rotation of the atomizing disk and the time taken for the process to re-establish a steady state. (The green line represents Dv10, blue line is Dv50, orange is Dv90, red is transmission)

size of the resulting droplets increases, as expected. However, after each change, it takes a certain amount of time for the particle size to steady at a new a value. The realtime data accelerate the assessment of different operating conditions but, at the same time, clearly highlight the need to run for around 30 minutes to generate robust particle size information at each new speed. Equally importantly, these data provide information as to when a process change in a manufacturing plant will feed through to a changed product that can be securely collected as in-specification material. The "sweet spot" for this process is found to be a speed of rotation of 8,000 rpm, which produces particles with the required Dv50 and an acceptable level of fines as quantified by Dv10 data (Table 2).

A modern approach

Spray-dried powders are prized for their fluidity, shape and tight particle size distribution. Producing standardized, spherical particles is crucial and relies on exerting effective process control. Timely, relevant particle characterization is essential. Commercial realtime particle sizing technology makes it possible to continuously monitor the quality of spray-dried powders, as they are produced, to drive rigorous process optimization and, ultimately, automated process control. Beyond size measurement, automated imaging delivers statistically relevant shape distribution data in minutes, providing further insight for precise control of the properties of spray dried powders. Together, these techniques support the effective application of spray drying technology and its exploitation to manufacture consistent products with well-defined properties and high value. ■

Edited by Scott Jenkins

TABLE 2. ATOMIZATION DISK ROTATION SPEEDS AND PARTICLE PROPERTIES

	10,000 rpm	8,000 rpm	6,000 rpm
Dv10 (μm)	23.57	32.22	38.98
Dv50 (μm)	44.71	61.38	71.78
Dv90 (μm)	80.44	112.45	122.68
Span	1.27	1.31	1.17

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Crossflow Membrane Filtration Essentials

Several aspects of crossflow membrane filtration , including process design, equipment selection and control, are detailed here

Alan Gabelman
Gabelman Process Solutions

IN BRIEF

- ADVANTAGES AND DISADVANTAGES
- BATCH PROCESS FLOWSHEET
- MF AND UF BASICS
- PRESSURE EFFECTS AND POLARIZATION
- DESIGN CONSIDERATIONS
- MEMBRANE MATERIALS
- MODULE CONFIGURATIONS
- DIAFILTRATION
- CONTINUOUS OPERATION
- MEMBRANE FOULING AND CLEANING

A membrane, also referred to as a semipermeable membrane, is a thin layer of material that selectively passes one or more components of a feed solution or slurry, while retaining the others. Biological membranes have existed since the dawn of time, but synthetic membranes are of greater industrial importance. These were first employed commercially in crossflow filtration operations in the 1960s, and growth in the ensuing years was remarkably fast. Today, crossflow membrane filtration (CMF) is a major unit operation that is pervasive in numerous industries.

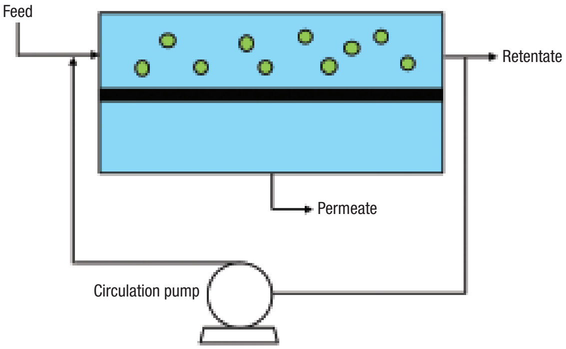

FIGURE 1. Crossflow membrane filtration (CMF) differs from conventional filtration in that feed flow is parallel to, rather than perpendicular to, the filtration surface

The concept is illustrated in Figure 1. Unlike conventional filtration [1], feed flow is parallel to, rather than perpendicular to, the filtration surface. During a given pass, only a small portion of the feed permeates the membrane and becomes permeate, while a much larger portion is retained as retentate. Most of the retentate is returned for multiple passes, by the action of the circulation pump. This allows a high linear velocity, which imparts a shear to the membrane that helps to keep the filtration surface clean. The valuable stream may be the permeate, the retentate, or both.

As shown in Table 1, membrane pore size varies considerably, with separation capabilities from angstroms (Å) to several microns (µm) in particle size. The relatively coarse microfiltration (MF) membranes are used to separate liquids from undissolved solids by size exclusion, as in traditional filtration processes. In principle, the liquid composition does not change, although in some cases there are phenomena other than size exclusion that do lead to retention of dissolved species, including adsorption onto the membrane surface or the undissolved solids themselves. The relatively large pores characteristic of microfiltration membranes are known as macropores.

Like microfiltration, ultrafiltration (UF) operates using traditional size exclusion, but with smaller mesopores rather than macropores. While both UF and MF are used to remove undissolved solids, UF membranes are also capable of separating large and small molecules in solution. UF membranes are described by their nominal molecular weight (MW) cutoff (NMWC), which is a rough indication of the smallest molecule that the membrane will retain. However, because

TABLE 1. CROSSFLOW MEMBRANE FILTRATION PROCESSES

Membrane process	Separation mechanism	Separation capability	Transport regime
Microfiltration	Size exclusion	0.1–10 µm	Macropores
Ultrafiltration	Size exclusion	MW 1,000–500,000 g/mol	Mesopores
Nanofiltration	Size exclusion, electrostatic exclusion	MW 100-1,000 g/mol	Micropores
Reverse osmosis	Solution/diffusion	MW <100 g/mol	Molecular
Gas separation	Solution/diffusion	Molecular size ~0.0001 µm	Molecular

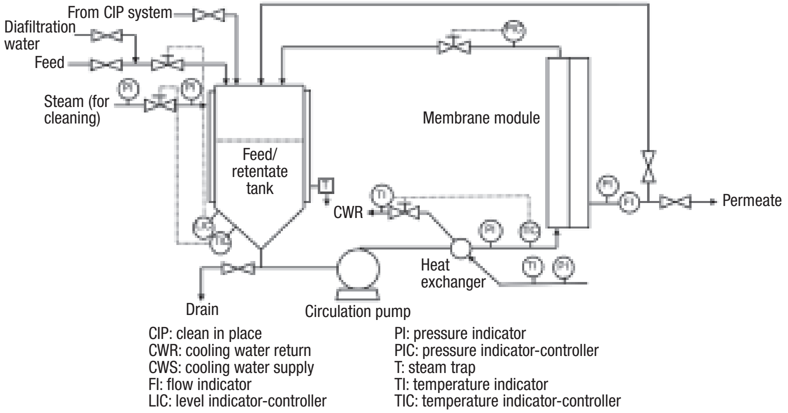

FIGURE 2. **Batch or semi-continuous crossflow membrane filtration processes can vary widely in complexity, depending on the application**

permeability depends on more than just molecular size, exclusion of all molecules above a certain NMWC is unlikely. As a rule of thumb, for complete removal of molecules of a given size, a membrane rated one-tenth that size should be selected. Ultrafiltration membranes are available with NMWCs of 1,000 to 500,000.

This article focuses on MF and UF, except for a brief mention of the other processes listed in Table 1. Nanofiltration membranes contain micropores that are tight enough to separate very small molecules, such as monosaccharides from disaccharides. For neutral molecules, the principle is size exclusion, the same as in UF and MF, with NMWCs of 100–1,000. However, nanofiltration membranes can be functionalized with charged groups, making electrostatic interactions an important factor in performance. Such membranes are used, for example, to repel anions in wastewater treatment applications. Nanofiltration is a relatively new classification — before the term was coined, these products were considered loose reverse osmosis membranes.

Reverse osmosis membranes were the first to see large-scale industrial use, primarily for desalination of seawater. Unlike the membrane processes discussed so far, the separation mechanism is solution/diffusion, meaning molecules dissolve in the polymeric membrane, then pass through by diffusion. In desalination, the most common application, the electrostatic attraction between ions and surrounding water molecules leads to hydrated structures that dissolve and diffuse much more slowly than free water molecules. For this reason, the free water passes through, while the ions are largely rejected. RO membranes are non-porous, meaning there are no distinct pores that accommodate fluid flow. The openings are the interstitial spaces between the polymer chains, and the transport regime is known as molecular because the size of these openings is of the same order of magnitude as the small molecules (nominally, molecular weight less than 100 g/mol) that pass through these membranes.

Finally, the tightest membranes are those used for gas separations — for example, separation of air into nitrogen and oxygen. These non-porous membranes also fall into the molecular transport regime, and pass molecules on the order of 0.0001 µm in size.

Advantages and disadvantages

The main advantage of crossflow membrane filtration is the shear imparted to the filtration surface, which reduces fouling by particulate matter or retained molecules. Another advantage is ease of scaleup, which is linear and straightforward. Pilot work is done with one or a few modules identical to those to be used in production, which minimizes scaleup risk. The required number of production modules is easily calculated from the target production rate and the permeate flowrate per unit filtration area, or flux, obtained in the pilot plant. Other advantages of CMF are: mild processing conditions (temperature, pressure); high product yield; ability to operate as a closed system, reducing risk to personnel and the environment when handling hazardous materials; and the ability to tailor membrane properties to match specific user requirements.

On the downside, the capital cost of membrane plants is relatively high, especially if exotic membrane materials are required. In addition, many polymeric membranes are subject to swelling in the presence of high concentrations of organic compounds, and cannot be operated at high temperatures. These challenges can be met with inorganic materials or high-performance polymers, but the cost is higher. Other disadvantages include formation of a polarization layer on the membrane surface, which reduces flux; fouling, requiring periodic cleaning; and in some cases, significant dilution when retained species are washed to increase product yield or remove contaminants — a process known as diafiltration.

Batch process flowsheet

A process flowsheet for a batch or semi-continuous membrane filtration process (continuous processes are discussed later) is shown in Figure 2. Please note that this is just an example. Like any chemical engineering unit operation, CMF processes vary widely in complexity, ranging from completely manual to highly automated. In a batch process, retentate is circulated through the membrane module then back to the feed/retentate tank, where the volume gradually decreases, and the concentration of the retained species increases, as permeate is removed. In a semi-continuous process, fresh feed is added to the tank at the same rate permeate is withdrawn, so that the retentate concentration still increases while the tank level remains constant.

The pressure driving force, known as transmembrane pressure (TMP), is generated by the combined action of the circulation pump and the

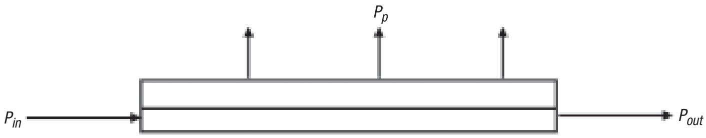

FIGURE 3. The pressure profile within a CFM module is an important factor to consider. Here, P_{in} is the feed/retentate pressure at the module inlet, P_{out} is the feed/retentate pressure at the module outlet and P_p is the permeate pressure

throttling valve downstream of the module. These must be balanced to maintain a stable operation. For example, throttling the valve will increase the transmembrane pressure, which tends to increase the flux (up to a point — this is discussed further later in the article). At the same time, the flowrate will decrease, and the reduced shearing action will result in a lower flux.

Note that in Figure 2, the pump discharge is cooled. This may seem counterintuitive, because in most cases, a higher temperature results in a lower viscosity, and in turn, a higher filtration rate. However, the circulation pumps used in crossflow membrane units are rather large, and generate a significant amount of heat. For this reason, cooling may be needed to avoid damage to the product, or in the case of common polymeric membranes, remain below the maximum allowable operating temperature. This is especially true in a batch process when the retentate volume becomes small. Cleaning is done without the need for disassembly, a feature known as clean in place (CIP), which is common in food and pharmaceutical applications. The indicated ability to recycle permeate to the feed/retentate tank is useful during certain portions of the CIP cycle.

MF and UF basics

Microfiltration membranes, which are used to produce clear permeate from slurries containing solids as large as 10 µm, typically have ratings of 0.1, 0.2, 0.45 or 0.65 µm. Required transmembrane pressure is low, usually 10–50 psi — as explained later, higher is not necessarily better. Volume reduction is typically 5–12, corresponding to undissolved solids of 100–700 g/L in the retentate. Greater volume reduction and higher retentate solids are achieved with rigid, incompressible solids than with soft or gel-like materials that are more difficult to handle. A partial list of applications of crossflow membrane microfiltration (CMMF) is given in Table 2.

In addition to the general advantages of CMF cited previously, CMMF offers a permeate that is completely solids-free, and typically more clear than filtrates from conventional filters [*1*] or centrifuges [*2*]. Moreover, no filter aids or other processing aids are needed. Disadvantages are the inability to produce a cake, as well as possible pluggage of membrane pores with compressible solids. However, if macromolecules are not needed, the latter issue can be overcome with the use of a coarse ultrafiltration membrane, with pores large enough to pass all but the largest dissolved molecules, yet sufficiently small to exclude undissolved solids. This technique is used, for example, in the filtration of apple juice.

Operating pressures in ultrafiltration are higher than in CMMF, but still relatively low. If undissolved solids are not present, volume reductions of 30 or higher are possible, much higher than those achievable with CMMF. The limitation may be the retentate viscosity, the solubility limit of one or more components or the practical problem of equipment holdup — that is, enough retentate is needed to fill the pipes and avoid pump cavitation. Ultrafiltration is widely used in the food industry — for example, to concentrate proteins in skim milk or cheese whey. The process is also commonly used in drug purification and other pharmaceutical applications, as well as water purification and chemical recovery in the textile, paper and waste-treatment industries.

Pressure effects and polarization

As with normal pipe flow, friction causes a loss in pressure from the inlet to the outlet of the module on the feed/retentate side. Referring to Figure 3, this pressure drop due to bulk fluid flow is expressed as in Equation (1) below:

$$\Delta P_b = P_{in} - P_{out} \tag{1}$$

This is not to be confused with transmembrane pressure, which is the difference between the average feed/retentate and permeate pressures, as shown in Equation (2):

$$\Delta P_{tm} = [(P_{in} + P_{out})/2] - P_p \tag{2}$$

Often, the permeate pressure is close to atmospheric, just high enough to overcome the friction loss in the downstream piping. Intuition tells us that a higher TMP results in a higher flux, and this is generally the case, up to a point. However, above some threshold value — typically about 1 bar — a polarization or gel layer forms on the membrane surface [*3*]. This layer is composed of undissolved solids and retained molecules in the feed. Moreover, because retained species concentrate near the membrane surface, precipitated components may be present if their solubility limit is reached.

Once the polarization layer is established, a further increase in TMP does not increase the flux, and may even reduce it. In addition, the polarization layer actually does the filtration, and the membrane itself has little effect on the permeate composition. To minimize the effect of the polariza-

TABLE 2. PARTIAL LIST OF CMMF APPLICATIONS

- Removal of cells or cell fragments from fermentation broth
- Removal of microorganisms from potable water
- Removal of microorganisms from milk
- Removal of fat from milk
- Removal of haze from gelatin
- Removal of µm-size particles from a variety of liquid streams
- Concentration of fine solids
- Pretreatment for ultrafiltration, nanofiltration or reverse osmosis
- Sterilization in the pharmaceutical industry
- Purification of therapeutic proteins and other pharmaceuticals
- Clarification of beverages (such as beer, wine or fruit and vegetable juices)
- Purification of fluids for semiconductor manufacturing
- Separation of radioactive solids from liquid waste in nuclear power plants

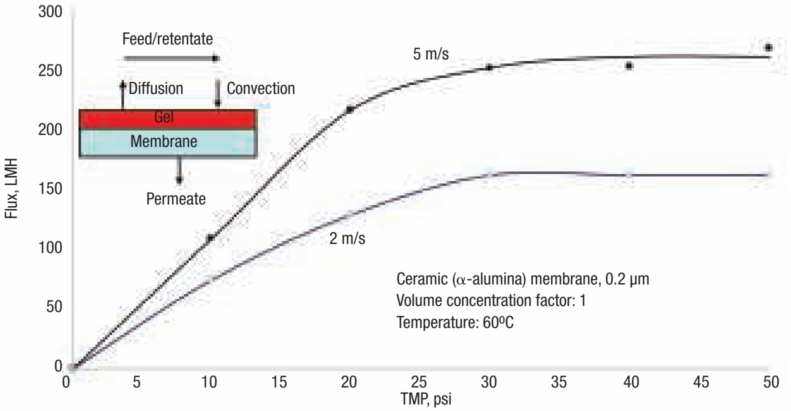

FIGURE 4. The flux (in liters of permeate per square meter of membrane area per hour, or LMH) versus transmembrane pressure (TMP) for microfiltration of corn starch hydrolysate at two different linear velocities is shown [*4*]

tion layer and maximize the flux, TMP should be kept relatively low — typically 10–15 psi for MF, and up to 50 psi for UF. For applications that are particularly sensitive to fouling, much lower TMP values are used, as low as 1–2 psi. These low TMPs require backpressure on the permeate side, an exception to the general practice of operating with the permeate near atmospheric pressure.

Evidence of the polarization layer is clear in Figure 4, which shows flux versus TMP for microfiltration of corn starch hydrolysate at two different linear velocities [*4*]. These data were collected at a concentration factor of one, meaning all permeate was recycled. At low values of TMP, the flux increases linearly with TMP, as one might expect. However, the curves begin to level off at about 25 psi, and above 30 psi there is no increase in flux upon further increase in TMP. Note that the maximum flux is higher at the higher linear velocity, because the higher shear reduces the thickness of the polarization layer, and in turn, the resistance to flow.

Membrane operation with an established polarization layer is illustrated in the inset in Figure 4. The retained species is transported from the bulk feed/retentate to the polarization layer by convection. The concentration of the retained species in the polarization layer increases, and this gradient drives diffusion back into the bulk. At steady state, the rates of transport to and away from the polarization layer are equal, and the polarization layer is stable. An increase in pressure causes an increase in the rate of convective transport to the layer, which leads to an increase in the retained species concentration, and in turn, increased rate of diffusion back into the bulk. A new steady state is established, and the net result is no change in flux, consistent with Figure 4.

Design considerations

Important design considerations include not only most or all of the considerations pertinent to the design of traditional filter or centrifuge operations, but also some that are unique to membrane systems. Key factors are described in the following paragraphs.

Productivity target (usually expressed as annual throughput). As with any chemical engineering unit operation, this figure forms the basis of membrane process design and economics. For example, throughput drives the choice of batch or continuous processing, with larger volumes making the latter more cost effective. Once the permeate flux is known from pilot work, the membrane area needed to reach the productivity target is a straightforward calculation, as mentioned above.

Fluid physical properties (especially viscosity). In most cases, flux decreases with increasing viscosity. For this reason, membrane processes are sometimes operated at elevated temperatures, with due consideration of the heat generated by the circulation pump. As discussed below, membranes made from inorganic materials and certain polymers allow operation at temperatures well above the limits of traditional polymeric membranes. However, the rate of membrane fouling may also increase with temperature, and this can negate some or all of the benefit. In addition to the change with temperature, any increase in viscosity with retained species concentration must also be taken into account.

Fluid composition. The starting concentration and solubility of retained molecules clearly have an impact on design — for example, they affect the achievable retentate concentration and, barring other limitations, volume reduction. If undissolved solids are present, not only is their concentration important, but also the nature of those solids. As with conventional filtration, rigid, spherical particles are more easily removed than ones that are soft, gelatinous or odd-shaped. Interactions between the feed and the membrane material also warrant consideration. For instance, components of interest can be lost if adsorbed onto the membrane surface. In this case, the designer must either select a membrane material with little affinity for the solute, or if possible, change the processing conditions (for example, pH) to reduce affinity.

Fouling tendency. Membrane cleaning adds cost, attributable not only to the required chemicals, but more importantly, to the associated downtime. Membrane materials, operating conditions, cleaning protocol, and where possible, feed properties, must be selected to minimize fouling, and in turn, cleaning time and frequency. While the feed composition gives some clues about expected fouling tendency, longterm pilot testing is needed to develop a full understanding.

Modules. The module specifications are a key part of membrane system design. Available module configurations are discussed in subsequent sections of the ar-

TABLE 3. FACTORS INFLUENCING MEMBRANE FLUX

Factor	General effect
Temperature	Higher temperature leads to higher flux, but rate of fouling may also increase
Undissolved solids concentration	Flux tends to decline with increasing solids content
Nature of undissolved solids	Flux and achievable volume reduction are higher with rigid, spherical solids than with soft, gelatinous or odd-shaped ones
Dissolved solids concentration	Flux tends to decline with increasing solids content, at least partly due to higher viscosity
Viscosity	Higher viscosity results in lower flux
Transmembrane pressure (TMP)	Flux increases with TMP up to a point, then further TMP increases have little effect
Linear velocity	Flux increases with linear velocity, but again up to a point
Pore size	Flux is usually higher with larger pores. One notable exception is with soft particulate matter, which may plug larger pores but are excluded from smaller ones
Porosity	A higher porosity leads to a greater flux, but mechanical strength is insufficient when porosity is too high
Membrane thickness	A greater thickness results in a lower flux

S. Koseoglu, Filtration and Membrane World LLC

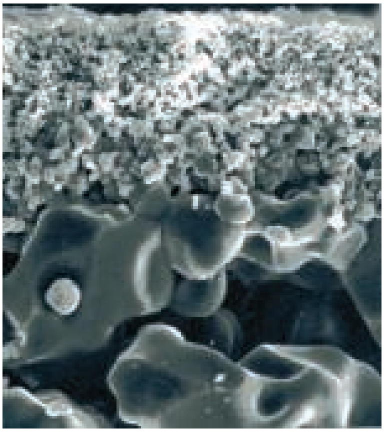

FIGURE 5: Asymmetric membranes, such as the one shown in this electron micrograph, are characterized by a thin selective layer on top of a more open support layer

ticle. Specifications include module length, diameter, size and location of ports, internal dimensions, materials of construction, and of course, membrane filtration area. Key operating specifications are temperature, transmembrane pressure and linear velocity. The latter is typically 1–2 and 5–7 m/s for polymeric and inorganic membranes, respectively. The benefit of the increase in flux with linear velocity (shown, for example, in Figure 4) is at least partially offset by the higher pumping cost. Thin channels offer higher membrane area per unit volume (that is, packing efficiency), but these are not suitable for streams with high viscosity or undissolved solids content. Those situations require wider channels, and in turn, lower packing efficiency.

Flux. Clearly, an important number in membrane system design is the flux, usually expressed as liters of permeate per square meter of membrane area per hour (LMH), or gallons per square foot per day (GFD). A similar performance measure is permeability, or flux per unit transmembrane pressure — for example, LMH per bar. Factors influencing flux and permeability are listed in Table 3. The goal of pilot testing is to quantify these effects, particularly those of transmembrane pressure, linear velocity and retained species concentration.

Membrane materials

In the early days of crossflow membrane filtration, most membranes were made from cellulose acetate, and this polymer is still used. However, its chemical resistance is limited, pH tolerance is only in the range from about 2 to 9, and the maximum allowable temperature is relatively low at 35°C. Since the advent of membrane filtration on an industrial scale, there have been considerable advances in polymer technology. Today, there are a variety of robust polymeric membranes that can operate at pH values ranging from 1 to 14, and in some cases, at temperatures of 120°C or even higher. Examples include polysulfone (PS), polyethersulfone (PES), polyvinylidene fluo-

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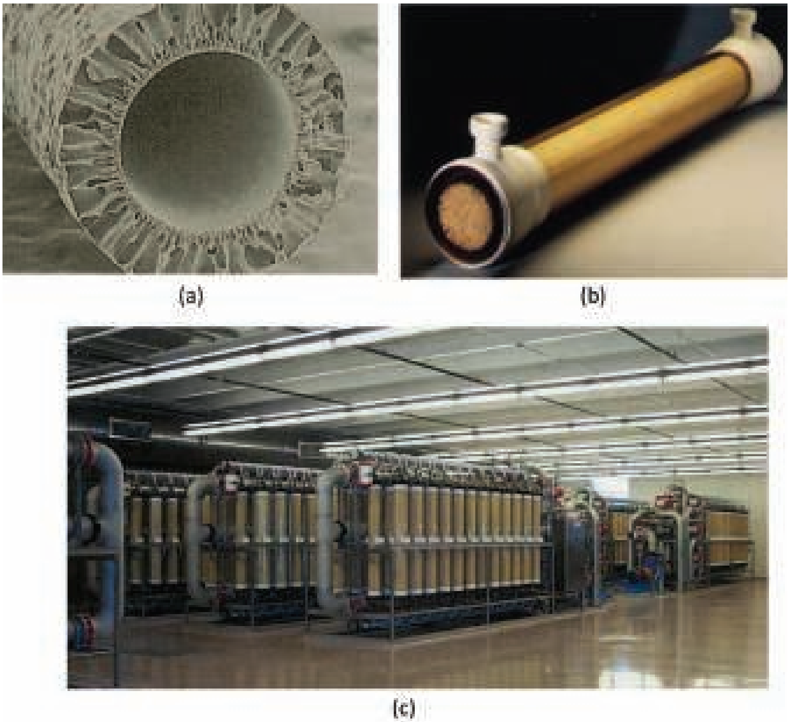

FIGURE 6. The hollow-fiber membrane configuration is shown as follows: (a): fiber cutaway displaying asymmetric pore structure; (b): module containing polysulfone fibers encased in a shell that is also polysulfone; (c) commercial unit using the modules shown in (b)

ride (PVDF), polyacrylonitrile (PAN), polyamides, polytetrafluoroethylene (PTFE) and polypropylene (PP).

Most of these membranes are asymmetric, characterized by a thin selective layer on top of a more open support layer (Figure 5). The tight selective layer, also called the dense, active or skin layer, does the actual filtration. Once this is done, a tight structure is no longer needed, and use of an open support minimizes the resistance to flow, while still providing the required mechanical integrity. A sublayer of intermediate pore size is also used in some cases. There are also symmetric membranes that contain pores with a uniform cross-section. These offer greater mechanical strength and longer life than asymmetric membranes, but the flux is lower.

Inorganic membranes are also available. These include ceramics, such as α-alumina, zirconia and metal oxides (notably, titanium dioxide), sintered stainless steel and graphite. Inorganic membranes are more robust than most polymers, able to withstand strong acids, bases and other corrosive chemicals, as well as high temperatures and pressures. Such membranes are particularly useful for high-fouling applications that require aggressive cleaning. Inorganic membranes are more expensive, but they last longer than polymeric ones, justifying the higher cost in some cases. Additional detail is given in the discussion of membrane modules presented below. Selection criteria for the membrane material are cost, compatibility with the process stream, any interactions (for example, adsorption) with feed components, ability to clean and expected lifetime.

Module configurations

Available module configurations are described in the following paragraphs, and a summary is presented in Table 4.

Hollow fiber. This module contains a bundle of polymeric tubes, also called hollow fibers, potted at both ends and encased in a shell. The geometry is analogous to that of a shell-and-tube heat exchanger, and the tube wall is the membrane. Feed enters the tubes at the bottom, retentate leaves at the top, and permeate passes through the tube walls, then exits through the shellside. Fibers are available in diameters of 0.25–6 mm, but 1–3 mm fibers are most common. A typical module is 10–20 cm in diameter by 1–1.6 m in length. The single fiber cutaway in Figure 6a clearly shows the asymmetric nature of the pores in the tube wall. The module shown in Figure 6b contains polysulfone fibers encased in a shell that is also polysulfone, and a commercial unit using these modules is depicted in Figure 6c.

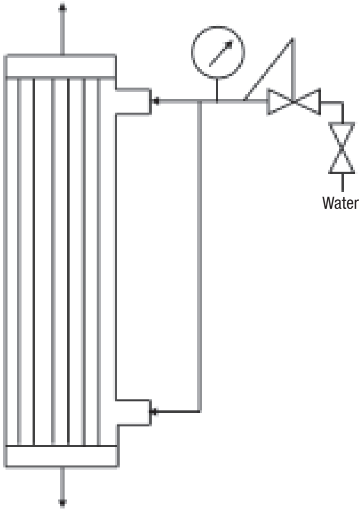

FIGURE 7. The ability to backwash hollow-fiber membrane modules provides more effective cleaning

The main advantage of the hollow-fiber configuration is the ability to tightly pack the fibers, allowing a high membrane area per unit volume, as high as 16,000 m^2/m^3. Another advantage is the ability to backwash for more effective cleaning. Backwash, shown schematically in Figure 7, entails forcing water through the membrane in the direction opposite to normal flow (that is, from the permeate to the feed/retentate side), to dislodge foulants from the membrane surface. These advantages make the hollow-fiber design an economical option for many applications, especially in the food and pharmaceutical industries. For example, hollow fibers are used in over 90% of the 1,500 wine filtration plants throughout the world.

A disadvantage of hollow-fiber modules is their greater fouling tendency compared to other module configurations. In addition, fiber pluggage can occur if large solids are present, a problem that can sometimes be resolved by prefiltration of the feed. If a larger fiber diameter must be used, there is a penalty in membrane area per unit volume. Finally, hollow fibers are not suitable for streams with high viscosity or undissolved solids content. Those applications are a better fit for a tubular module, discussed below.

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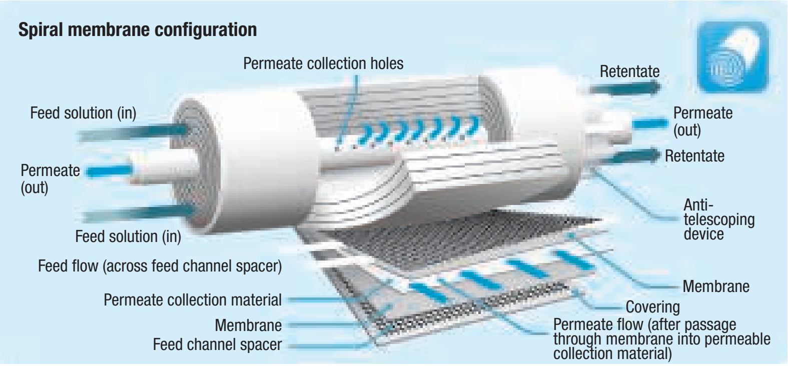

FIGURE 8. Spiral-wound modules are commonly used for water filtration

Spiral wound. As shown in Figure 8, this design consists of one or (usually) more membrane envelopes wound around a perforated central core. Each envelope contains two rectangular membrane sheets facing away from each other, separated by a porous spacer. These spaces provide a path for the permeate, which flows around the spiral to the central core, then exits the module. The spaces between adjacent envelopes, separated by grids, serve as flow channels for the feed/retentate. Each envelope is sealed with adhesive on three sides, and the fourth side is attached to the central tube. Typical channel width, module diameter and length are 0.25–0.5 mm, 10–40 cm and 1–1.5 m, respectively.

Spiral-wound modules are commonly used for water purification, and the high volume drives the cost down for other applications as well.

TABLE 4. MODULE CONFIGURATIONS

Configuration	Description	Advantages	Disadvantages	Additional comments
Hollow fiber (Figure 6)	Fiber bundle potted at both ends, encased in a shell	• High filtration area per unit volume • Can be backwashed	• High tendency to foul • Fibers can plug • Not recommended for high viscosities or undissolved solids contents	Often an economical option for low-viscosity, low-solids feeds
Spiral wound (Figure 8)	Membrane envelope(s) wound around a central permeate collection tube	• Prevalence in water purification drives cost down for other applications as well • Low energy consumption • High filtration area per unit volume	• Not suitable for fibrous solids • Not recommended for high viscosities or undissolved solids contents • Flow channels can plug • Not recommended for high-value products because modules do not drain completely • Complex design and structure • Cannot be backwashed	Most common and economic configuration in use today. Often a good choice for low-viscosity feeds with low levels of non-fibrous solids
Tubular (Figure 9)	Active layer is deposited onto a porous tubular support, which is situated in a pressure vessel housing	• Resistant to fouling because of good fluid hydrodynamics (turbulent flow) • Easy to clean • Able to handle high viscosities, high undissolved solids contents and large particles	• Low packing density leads to high cost per unit area • High energy consumption • Usually cannot be backwashed	Good choice for streams with high viscosity, elevated concentration of large solids, or high fouling tendency
Plate and frame (Figure 10)	Flat sheet membranes assembled in a plate-and-frame device reminiscent of a filter press	• Baffles can be used to achieve high fluid velocities at lower circulation rates than with other designs • Can be built with wide plate spacing, to accommodate high viscosities or undissolved solids levels	• Low area per unit volume • High cost of frames drives overall cost up	In general, these are being replaced by other configurations
Ceramic (Figure 11)	Porous ceramic monolith containing parallel flow channels, with active layer deposited onto the channel surface	• Can withstand high temperatures. • Highly resistant to chemical attack • Handle high linear velocities without excessive abrasion • Long membrane life	• Low area per unit volume • High capital cost (but may be justified by longer life) • High pumping cost	Ultimate in tolerance of severe process conditions and resistance to chemical attack
Sintered stainless steel (Figure 12)	Sintered stainless-steel tubes with TiO_2 active layer deposited onto the surface, encased in a stainless-steel shell	• Same as for ceramics, but with lower cost • No seals to fail because channels are welded in place	• Available pore sizes are limited • Stainless steel is vulnerable to chloride stress corrosion • TiO_2 active layer may be subject to abrasion	A lower-cost alternative to ceramics for some applications

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(a)

(b)

FIGURE 9. Tubular modules employ a shell-and-tube design, as shown in (a), a module containing PVDF tubes and a polysulfone shell; (b) shows a commercial unit using the modules shown in (a)

Additional advantages are lower energy consumption compared to other designs, and like hollow-fiber modules, high area per unit volume. On the downside, spiral-wound units are not suitable if fibrous solids are present, because these will clog the grids. Prefiltration is recommended to remove particles larger than one twentieth of the channel width (1/20 rule). Moreover, as with hollow-fiber modules, process streams with high viscosity or high undissolved solids content are better suited for tubular modules. Because spiral-wound modules are difficult to drain completely, the hollow-fiber design is preferred for valuable products. Finally, the design and structure of spiral-wound modules are complex, and they cannot be backwashed.

Despite these disadvantages, the spiral-wound module is the most common configuration in use today. It is the most economical choice for many applications, particularly water purification, as mentioned previously.

Tubular. Like the hollow-fiber design, tubular modules have a shell-and-tube geometry, but the tubes are larger, with the inner diameter ranging from 2.5 to 25 mm. The tubes consist of a porous support, such as fiberglass-reinforced epoxy, with the active layer formed on the inside surface. The shell is made from stainless steel or a hard polymer. A single module and an industrial unit are shown in Figure 9.

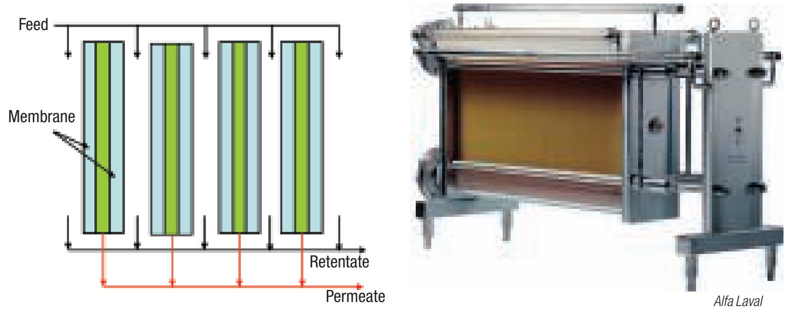
Tubular modules are chosen for feeds that are difficult to handle, and are not suitable for hollow fiber or spiral wound designs. These include feeds with high viscosity, high undissolved solids content, large particles (up to 2.5 mm; prefiltration: 1/10), or highly compressible or gelatinous solids. Advantages — in addition to the ability to handle these challenging streams — are high resistance to fouling, attributable to good fluid dynamics (turbulent flow); ease of cleaning; and simple module design and structure. Disadvantages are low packing density, and in turn, low area per unit volume; high energy consumption; and the inability to backwash. Applications of tubular modules include wastewater treatment, paint recovery and juice clarification.

Plate-and-frame modules. This is one of the earliest designs. These modules contain a series of flat membrane sheets, usually disk-shaped, arranged in a plate-and-frame assembly. As shown in Figure 10a, the membrane side of each sheet faces a feed channel, with the space between the opposite sides providing a flow path for permeate. This assembly, reminiscent of a traditional filter press, is situated between two end plates, with gaskets placed as needed to direct flow. A commercial unit is shown in Figure 10b.

Feed channels can be narrow, for non-viscous, low-solids feeds, or wide, to accommodate more difficult process materials. In addition, baffles can be used to reach high velocities at low pumping rates. On the downside, the low area per unit volume and high cost of frames have led to a decline in the popularity of plate-and-frame modules.

Ceramics. A ceramic module consists of a porous ceramic material, called a monolith, through which parallel flow channels have been formed. The separation is performed by a thin active layer of smaller particles deposited onto the inside surface of the channels. Channel diameters are 2–6 mm, and pore sizes extend from 40 Å (NMWC = ~1,000) to the μm range. The active layer is usually α-alumina, zirconia or titanium dioxide, while the monolith is typically α-alumina or other metal oxide.

Examples of ceramic modules are shown in Figure 11. As with hollow-fiber and tubular designs, a shell-and-tube geometry is used. The cylindrical inserts in the photograph are known as elements; a common design is 19 channels per element.



Alfa Laval

(a)

(b)

FIGURE 10. A plate-and-frame membrane module is shown schematically (a); and as an industrial unit (b)

Atech Innovations Gmbh

FIGURE 11. Ceramic modules such as these can withstand higher temperatures and more aggressive chemicals than polymeric modules

In operation, permeate coming through the active layer flows by gravity to the bottom of each monolith, then into the shell space and out the exit port. Meanwhile, feed/retentate transverses the length of the channel, then leaves.

Ceramic modules can withstand temperatures of 150°C or more, much higher than the maximum of 50–55°C for many polymers. This is useful for viscous feeds, and allows high-temperature cleaning in heavily fouling applications. Moreover, ceramics are highly resistant to chemical attack, permitting their use with process streams that attack polymers. An example is the use of ceramic modules to remove wax from citrus oils, which are highly corrosive to most polymeric membranes [5]. Their chemical resistance also allows ceramics to be aggressively cleaned with strong acids, bases or other harsh chemicals.

Another advantage of ceramic modules is the ability to back-pulse, also called blowback. With this technique, a periodic pulse of permeate is delivered in the direction opposite to normal flow — that is, from the permeate to the feed/retentate side. This improves flux by dislodging foulants from the membrane surface. The concept is like backwashing, discussed earlier, but backwashing uses an extended flow of water during cleaning, while back-pulsing involves periodic pulses of permeate that are delivered while running product. Pulse frequency and duration vary, but typical settings are once every 2–5 min for 0.5 s.

Additional advantages of ceramic modules are higher abrasion resistance than polymers, allowing operation at a higher linear velocity; and membrane life up to 10 years, compared to 1 to 2 years for a typical polymeric membrane. The main disadvantage of ceramics is their

Graver Technologies

FIGURE 12. A sintered stainless-steel module is fabricated as one solid, highly porous unit

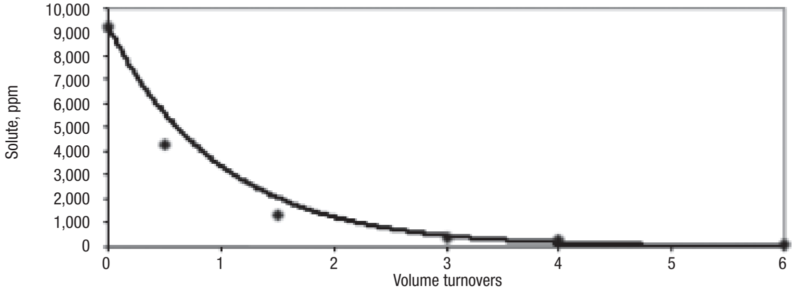


FIGURE 13. In this example, diafiltration is used for the removal of residual crosslinking agent from a slurry of polymer beads. The points are the actual data and the solid line is the prediction from Equation (3)

high cost, although this is sometimes economically justified by their longer life. Other disadvantages are low area per unit volume, part of the reason for the high cost; and high pumping cost, a downside of operating at a higher linear velocity.

Sintered stainless steel. These modules are made by placing powdered stainless steel in a mold, then heating to a temperature just below the melting point. Atoms diffuse across particle boundaries, fusing the particles together to create one solid, highly porous piece. As shown in Figure 12, again, a shell-and-tube geometry is employed. The active layer is titanium dioxide (TiO_2), which is annealed onto the inside surface of the channels. These modules offer most of the same advantages as ceramics, including high temperature tolerance and resistance to chemical attack, but at a lower cost. In addition, there are no seals to fail, because all connections are welded. On the downside, available pore sizes are limited with stainless-steel modules, stainless steel is subject to chloride stress corrosion, and erosion of the TiO_2 active layer can occur in some applications.

Diafiltration

As mentioned above, diafiltration is used to recover additional permeable components from the retentate, when the permeate is the valuable stream. Alternatively, when the retentate is valuable, diafiltration serves to improve purity by removing permeable contaminants. In a batch process (such as in Figure 2), diafiltration water is added at the same rate that permeate is removed, so that the rententate volume remains constant. It is easy to show that the displacement of permeable components obeys the exponential relationship given in Equation (3):

$$C(t)/C_0 = e^{-Wt/V} = e^{-N} \qquad (3)$$

Here, $C(t)$ is the concentration of the permeable component(s) at time t, C_0 is the concentration at the start of diafiltration, W is the flowrate of diafiltration water (equal to the permeate flowrate), V is the volume of retentate and N or Wt/V is the number of volume turnovers — that is, the volumes of diafiltration water per volume of retentate. For example, if the retentate volume is 1,000 L, each 1,000 L of diafiltration water added is one turnover. The data in Figure 13 are for removal of a residual crosslinking agent from a slurry of polymer beads. As predicted by Equation (3), 95% of the solute was removed after three turnovers.

When the permeate is the valuable stream, in most cases, the water added during diafiltration must be removed downstream, usually by evaporation. The optimum amount of diafiltration water represents a trade-off between the value of the recovered product and the cost of energy.

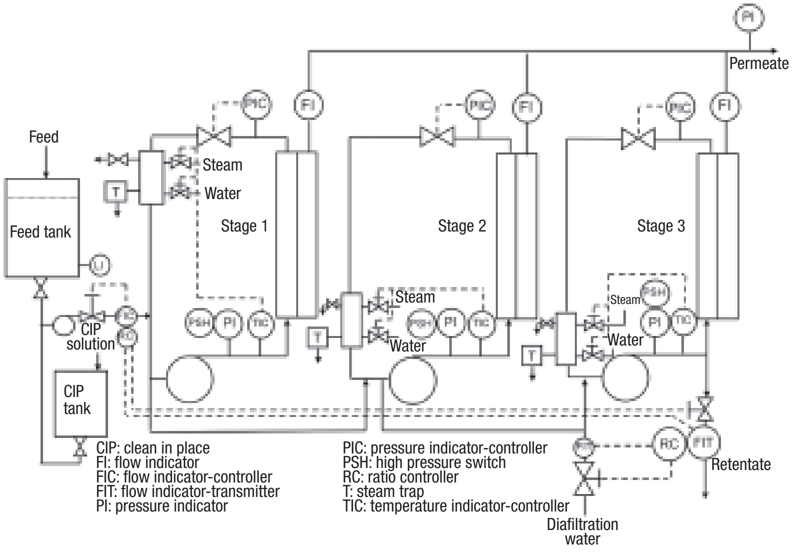


FIGURE 14. This example of a flowsheet for a three-stage, continuous crossflow membrane filtration process includes diafiltration added to the third stage

Continuous operation

Batch and semi-continuous processing are discussed above, and these operating modes are important when volumes are relatively small. However, as with other chemical engineering unit operations, continuous processing is more efficient and cost-effective when volumes are large. In most cases, continuous membrane filtration processes contain multiple stages. This is advantageous because, for many applications, flux declines with increasing concentration of the retained species. Since each stage operates at the flux corresponding to the concentration of the retentate leaving that stage, the overall flux is higher with multiple stages, and less membrane area is needed to reach the targeted productivity. A continuous

TABLE 5. COMMON CLEANING AGENTS

Type of cleaner	Example	What it removes
Caustic	1–2% NaOH, pH > 10	Proteins, microorganisms, biological debris
Detergent	0.1% Alconox	Particulate matter, miscellaneous organics, colloidal fouling
Enzyme-based detergent	0.1% Tergazyme	
Sodium hypochlorite bleach	200–1,000 ppm in water	Cleans and sanitizes
Acid	1–2% sulfamic acid	Inorganic scale

membrane system with an infinite number of stages requires the same filtration area as a batch unit. However, addition of stages eventually results in a diminishing return, because the cost of the additional circulation pump, piping, controls and other components exceeds the savings obtained from the reduced filtration area. In general, the area needed for five stages is within 20% of the batch area.

The continuous membrane filtration process shown in Figure 14 has three stages, with diafiltration water added to the third stage. With the control strategy employed, the feedrate is set independently, while the flowrate of final retentate is modulated by ratio control to maintain the desired volume reduction. Ratio control is also used to regulate the flow of diafiltration water, based on the retentate flowrate. Each stage has provisions for temperature control by heating or cooling, retentate back-pressure control and, to protect the module, a high-pressure switch that shuts off the circulation pump when activated. The circulation pumps are large, and the feedrate to each stage is only a small fraction of the circulation rate. Note that there are no controls regulating the flow of retentate from one stage to the next. These are not necessary because the system is self-adjusting and stable.

There are a number of strategies for minimizing the rate and extent of fouling. Prefiltration to remove large particulate matter, using a conventional filter or centrifuge, is sometimes helpful. Similarly, fouling can sometimes be reduced by upstream removal of large molecules with a tendency to foul, using a UF membrane with a relatively high NMWC.

Membrane fouling and cleaning

Because cleaning time is non-productive, productivity is maximized when cleaning is infrequent, fast and effective. To minimize the rate of fouling and specify an effective cleaning protocol, fouling mechanisms must be well understood. Fouling may be organic, inorganic or microbiological in nature. Usually, the foulants are present as physical buildup on the membrane surface, but they may also be adsorbed, or small particulate matter may penetrate the pores. Chemical reactions on the surface may also contribute to fouling.

There are a number of strategies for minimizing the rate and extent of fouling. Prefiltration to remove large particulate matter, using a conventional filter or centrifuge, is sometimes helpful. Similarly, fouling can sometimes be reduced by upstream removal of large molecules with a tendency to foul, using a UF membrane with a relatively high NMWC. Upstream dilution of feed can reduce the rate of fouling, with the downside that a larger volume needs to be filtered. Judicious selection of the membrane material is crucial to minimize interactions with the process stream that may lead to fouling. In some cases, such interactions can be reduced by changing processing conditions — for example, temperature or pH. Finally, a high linear velocity is needed to impart sufficient shear at the membrane surface.

Common cleaning agents are listed in Table 5. Typically, the cleaning protocol calls for several such cleaners in sequence. For example, the following steps may be used to clean a membrane handling a stream containing microorganisms and proteins, such as a fermentation broth:

- Water rinse
- Detergent or caustic cleaning
- Water rinse
- Acid cleaning
- Water rinse

A cleaning cycle such as this one may require 3 to 4 hours. Except when using acids, cleaning is usually more effective at elevated temperature. Acid cleaning is done at room temperature because the inorganic scale it is intended to remove (for example, calcium salts) often exhibits inverse temperature solubility. ■

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Gas-Phase Filtration Media Properties: What to Know

There are many physical properties that can impact the ultimate performance of adsorbent media in gas-phase filtration applications

Chris Muller
Purafil, Inc.

IN BRIEF

- MEDIA SHAPE
- MOISTURE CONTENT
- BULK DENSITY
- ADSORPTION EFFICIENCY
- REMOVAL CAPACITY

Gas-phase filtration media specifications may list numerous physical parameters, such as shape, moisture content, bulk density, impregnation level, size and hardness (crush strength or abrasion), as important determinants of final media performance. The way these physical characteristics can be controlled and optimized during media manufacturing processes can ultimately determine media performance when installed into any number of end-use applications, including standalone pressurization or recirculation units to prevent corrosion in pulp and paper mills or petrochemical plants, or in deep-bed scrubbers in petroleum refineries and wastewater treatment plants to contain a release of toxic chemicals.

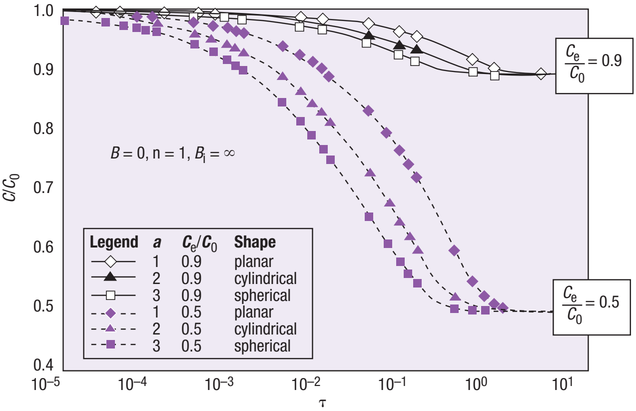

FIGURE 1. The effects of shape factor (*a*) and concentration on adsorption are shown for both theoretical and numerical solutions [*1*]

In order to optimize the packing density, whether the media is to be used in disposable modules or deep-bed scrubbers, spherical media is generally preferred. It is also the most favorable shape with regard to the diffusion of contaminants into pellets. This article discusses the physical parameters of media shape, moisture content and bulk density specifically as they relate to overall media performance, with some general discussion of related parameters.

Media shape

The ability of particles to fill space by packing together is obviously related to their shape. Spherical-shaped media of uniform size provide the most uniform packing, because with both planar (flake) and cylinder shapes, the media could settle upon itself. With spheres, one could ideally pack them to take up almost 75% of the available space. However, this would only occur with complete packing of the particles in the media container. It would not be achieved by simply pouring them into the container — rather, randomization effects will only yield a maximum packing fraction of 60–65%.

With flake and cylindrical shapes, the packing density from random packing methods, such as pouring without tamping or vibrating, would result in a packing fraction similar to that of spherical media. However, over time, both of these media shapes can stack upon themselves along their planar axes, resulting in a higher packing fraction and settling of the media. This leads to bypass and a reduction in performance of the air-cleaning system.

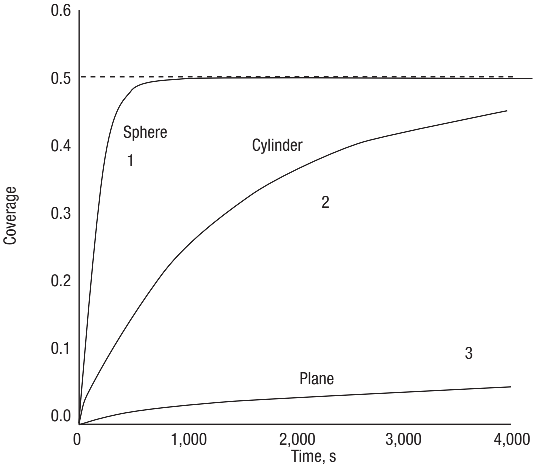

FIGURE 2. Different adsorbent geometries are compared for the same adsorption process. Here, coverage indicates the fraction of adsorption sites of the surface of the media unavailable versus the number of vacant sites still available for adsorption [2]

Spherical shapes allow more access to the external surface of the particle, which is important in adsorption and in taking advantage of the large internal surface area. When planar or cylindrical media "line up" or stack upon themselves, this effectively blocks access to the external surfaces in contact with each other. Proper packing of spherical media limits the surface area affected by particle-to-particle contact and assures access to the maximum number of adsorption sites.

Adsorption is usually described through isotherms — the amount of adsorbate (contaminants) on the adsorbent (media) as a function of its pressure or concentration at constant temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials. There are basically two well-established types of adsorption isotherms: the Langmuir and Freundlich adsorption isotherms.

Among numerous studies on adsorption dynamics of adsorbent media, little has been reported on the effect of shape. One study compared adsorption dynamic processes for three adsorbent shapes: spherical, planar and cylindrical. The results of a comparison of both Langmuir and Freundlich adsorption isotherms and kinetics showed that the rate of adsorption will increase as the shape changes from planar to cylindrical to spherical, indicating that there is a minimum of surface energy for the sphere among the three media shapes. In other words, the spherical geometry is the most favorable for adsorption, and the planar geometry is the most unfavorable.

There are no simple representations of the Langmuir and Freundlich adsorption models that can easily explain the changes in adsorption rates between spherical and cylindrical media shapes. The easiest thing to do is to provide a graphic that shows these differences.

Figure 1 [1] compares theoretical and numerical solutions for the rate of adsorption in terms of concentration in air (C), equilibrium concentration (C_e) and initial concentration (C_0), all in kg/m^3. Other pertinent terms in Figure 1 are the Freundlich coefficient (n), the dimensionless time (τ) and the dimensionless Biot number (B_i). Figure 1 shows that the rate of adsorption will increase as the adsorbent's shape factor (a) increases in all cases, indicating that there is a minimum of surface energy for the sphere among the three shapes of adsorbents.

Figure 2 [2] shows another representation of the effect of different media geometries (shapes) on adsorption rate. Additional graphs could be provided to show the relationships between the rates of adsorption (or removal) and other physical parameters, but each has the same basic shape. The spherical media is always to the left of the cylindrical media, indicating a faster adsorption rate.

The real power of the Langmuir and Freundlich adsorption models is in providing a reference framework within which one is able to compare the adsorption rates of spherical, cylindrical and planar geometries in a relative scale. Figures 1 and 2 show this very well.

Moisture content

Plain activated carbon used for air filtration typically has a moisture specification of 2–5 wt.%, and as long as the moisture content remains below 8–10 wt.%, the performance is relatively unaffected. Above a moisture content of 10 wt.%, carbon begins to preferentially adsorb water from the air, which occupies adsorptive sites and reduces the efficiency and capacity of the carbon. The water adsorption isotherm for activated carbon (Figure 3) shows that as long as the relative humidity (RH) of the airstream remains below 50–55%, the carbon performance should not be affected. The carbon will equilibrate with the moisture content of the air. However, this can produce periods of much lower performance for a great number of contaminants. This is especially so when the carbon is used to clean outdoor air.

Some engineered media can operate in a specified RH range of 5–95%, although it is recommended to operate in the range of 20–80% for optimum performance. Operation at the extremes of the RH range (for instance, <20% or >80%) can cause a drop-off in performance due to the following:

1. The media "drying out," which causes the chemical impregnant to come out of solution and not be available for reaction
2. The media becoming "soft" with too much water. Although a "soft" media still retains full functionality (and even works better in some applications), this can cause settling of the media due to abrasion loss, leading to bypass through the system

Bulk density

Practically all granular adsorbent or chemisorbent media are almost always sold by weight, and the overall removal capacity for any given air cleaning system is determined by the volume of media it holds. Although the media volume remains constant for a given system, the media weight can vary significantly due to differences in the media's bulk density. For the purposes of this article, the bulk density is defined as the mass (weight) of the media divided by the total volume it occupies. This total volume includes particle volume, inter-particle void volume and internal pore volume.

Let's say we are comparing two media (A and B) being offered for the same application. Media A has a bulk density of around 45 lb/ft^3 (0.721 g/cc). Media B has a bulk density of around 34 lb/ft^3 (0.545 g/cc). Based on this, and assuming an equal cost per pound of media, Media B would cost less than Media A to fill or replace the media in the same air-cleaning system. However, this is misleading and does not provide a true cost-of-ownership comparison. To do so requires a determination of the removal capacity of the media (in units of g/cc or wt. %).

Media B lists a removal capacity for hydrogen sulfide (H_2S) of 17 wt.%. With a media bulk density of 34 lb/ft^3, the volumetric capacity can be calculated as 5.78 lb of H_2S removed per cubic foot of media (34 × 0.17). Media A's stated H_2S removal capacity is 20 wt.%, which converts to 9 lb of H_2S removed per cubic foot of media (45 × 0.20). Comparing these media based on their bulk density and using the removal capacities provided, Media A exhibits a 50% higher volumetric H_2S-removal capacity. Air cleaning systems using granular media are filled by volume, not by weight, therefore, comparisons based on a volumetric capacity and not a weight capacity should be used when developing media changeout schedules and lifecycle cost estimates.

Adsorption efficiency

As mentioned in the discussion of media shape, the rate of adsorption is greater for spherically shaped media. This in turn can lead to a higher adsorption efficiency for a given combination of contaminant types and concentrations, and ultimately higher removal capacities and efficiencies. However, any discussion of adsorption efficiency must include an investigation of how freely contaminants can diffuse into the inner structures (surface area) of the media pellet.

When uniform-sized spheres are packed into a media container, the same cross-sectional area presents itself for each media pellet, promoting radial diffusion. In cylindrical-shaped media, both radial and longitudinal diffusion are important. Radial diffusion considers how long it takes materials to diffuse across the radius of the cylinder, whereas longitudinal diffusion considers how long it takes materials to diffuse

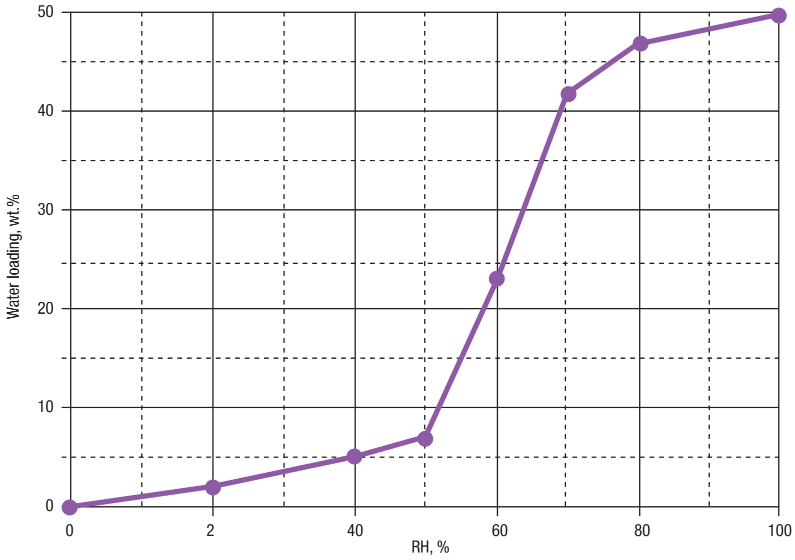

FIGURE 3. This water adsorption isotherm for activated carbon shows the effect of relative humidity (RH) on the adsorption performance

through the length of the cylinder. If the cylinder's length-to-diameter ratio approaches 1, the rate of diffusion approximates that of a sphere, which favors the more efficient radial diffusion.

With cylinder-shaped particles, the random nature of a packed bed causes both the radial and longitudinal axes to present themselves. When airflow is hitting the end of the cylinder, slower longitudinal diffusion is the result. With increasing cylinder length, the particles tend to line up along their length. This can have two results. If the ends of the cylinders are perpendicular to the air flow, longitudinal diffusion predominates. If the media particles are lined up along their length, radial diffusion dominates. However, this media configuration results in a high packing density, which reduces the void volume around the media, increasing the pressure drop, and decreasing the rate of adsorption.

Spherical media provides for more efficient adsorption due to the uniform radial diffusion of contaminant gases. The cylindrical shape of extruded media involves both radial and the less efficient longitudinal diffusion. Adsorption efficiency is significantly reduced when the cylinder length-to-diameter ratio exceeds 1.2. Many extruded media have an average length-to-diameter ratio of 1.75, based on numerous sample analyses.

Removal capacity

To reiterate the statement from above, gas-phase air cleaning systems using granular adsorbent media are filled by volume and not by weight. Therefore, one needs to look at the removal capacity of a particular media in terms of how much contaminant material a given volume of media can remove before it becomes spent and needs to be replaced.

Media manufacturers may report a volumetric removal capacity or as a percent by weight. These values are determined according to the test procedures described in the ASTM International Standard D 6646-01, "Standard Test Method for Determination of the Accelerated Hydrogen Sulfide Breakthrough Capacity of Granular and Pelletized Activated Carbon." Although the title may suggest that this test method has limited use, it is readily applicable for many different media types against a wide variety of contaminants.

Breakthrough capacity testing indicates how much the media can adsorb in grams of gas (for example, H_2S) per cubic centimeter of media regardless of the system. The volumetric capacity uses the media density (g/cc) and the removal capacity to determine the total amount of contaminant gas an air-cleaning system can remove per unit volume. This provides a truer comparison of one media to another, because differences in bulk density are taken into account.

Another helpful practice is to list a media's capacity for a single contaminant as a minimum value, because the specific conditions under which the media would be used cannot be assured. This provides assurance that the media will be able to remove a certain amount of contaminants before it has to be replaced. This also allows for the provision of media life estimates that do not exceed those experienced under actual use conditions. By determining removal capacities against individual contaminants and by specifying minimum values, users can properly account for the actions of other contaminants that might be present in the airsteam against the media. ■

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Cooling Towers: Estimate Evaporation Loss and Makeup Water Requirements

Applying mass and energy balance calculations yields critical operating insight

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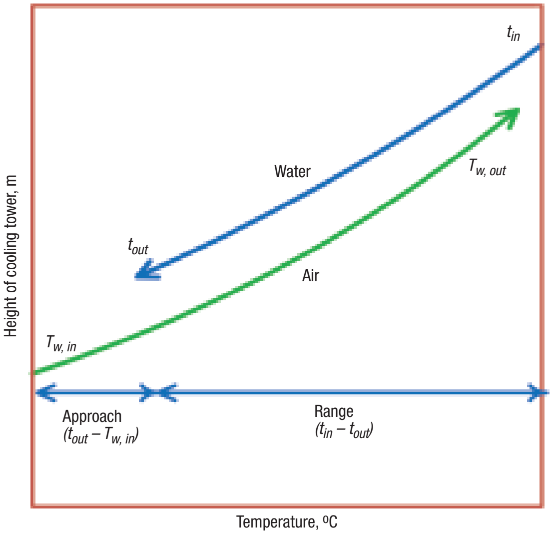
Cooling towers are an important unit operation in chemical process industries (CPI) operations. Applying mass and energy balance calculations enables process engineers to evaluate evaporation loss, blowdown and makeup-water requirements, and to evaluate the performance of the cooling tower. In this article, an illustrative study showcases an induced-draft cooling tower and describes several key parameters — range, approach and efficiency — and their significance. Two methods are discussed to estimate evaporation loss. Requirements for blowdown and makeup water are also detailed.

Cooling tower operation

The cooling of process streams and condensation of vapors are important functions in CPI operations. The use of a cooling tower is the most common way of extracting waste heat in CPI operations, and water is the most commonly used coolant to remove waste heat in the majority of such operations. A typical large petroleum refinery that processes 40,000 metric tons (m.t.) of crude oil per day requires 80,000 m^3/h of cooling water. This is roughly equivalent to 25 barrels of water for every barrel of crude oil processed [*1*].

In a cooling tower, the hot water stream (typically called the cooling water return) is introduced downward through spray nozzles into fills inside the tower. There are different types of fills — splash, trickle and film — that are aimed at creating more surface area, to maximize contact between the hot water stream and air. As air rises inside the tower, it receives the latent heat of vaporization from the water, and thus the water is cooled.

FIGURE 1. Shown here is the typical variation of the water temperature and the wet-bulb temperature of the air stream as the hot water inlet stream flows down from the top of the cooling tower and the air stream flows upward along the height of the cooling tower



As a rule of thumb, for every 10°F (5.5°C) of water cooling, 1% total mass of water is lost due to evaporation. The humidity level of the up-flowing air stream increases, and once it leaves the tower the air stream is almost saturated. The temperature profile of the water and the wet-bulb temperature of the air along the height of a typical cooling tower is shown in Figure 1.

The cooled water is collected in the sump (or basin) of the cooling tower, and it is typically pumped to the plant as the cooling-water-supply (CWS) stream. After extracting heat from the process units, this stream is returned to the cooling tower, as the cooling-water-return (CWR) stream. The heat load extracted from the process unit is finally released to the environment in the cooling tower. A cooling tower is designed to remove the total heat load that is extracted from the plant by reducing the CWR temperature to the CWS temperature.

Case study

The CWR line from the process unit enters an industrial cooling tower at 45°C and leaves at 33°C, as shown in Figure 2. The tower has three cells, each operating at 2,500 m^3/h of water flow. The total flow 7,500 m^3/h is measured at the CWR line. The dry-bulb temperature and wet-bulb temperature of the inlet air are measured as 30.3°C and 29°C, respectively. The dry-bulb temperature of the exit air is 41.5°C, and it is assumed to be 100% saturated. This case study is aimed at calculating the unknown variables — that is, evaporation loss, air flow through the tower, blowdown flow, and the required makeup water flow. First, the important parameters — approach, range and efficiency — are detailed.

Approach. The approach is defined as the difference between the water temperature at the tower outlet (t_{out}) and the wet-bulb temperature of the inlet air ($T_{w,in}$). The approach represents the cooling tower capability. In general, the larger the tower, the smaller is the approach. In this case study, the approach is 4°C.

Theoretically, the extent of maximum possible cooling that could be achieved through a cooling tower would be to produce a stream that is at the wet-bulb temperature of the ambient air. However, to achieve this theoretical maximum, the tower would need to have infinite height. So the practical limit of the CWS temperature is generally considered to be 4°C above the wet-bulb temperature of ambient air. For design purposes, the worst scenario — that is, the summer season wet-bulb temperature — needs to be considered.

Range (ΔT). The range is the difference between the water temperatures at the inlet and the outlet of the cooling tower ($t_{in} - t_{out}$). In this case, the range is 12°C. The range does not represent the cooling tower capability; rather, the range is based on the cooling-water circulation flowrate (L_{in}), and the sum of the heat loads taken from the heat exchangers in the process unit (Q), and it is not related to the size or capability of the cooling tower. On the other hand, an increase in range will cause an increase in approach, if all other conditions are not changed. The range is shown in Equation (1):

$$\Delta T = \frac{Q}{L_{in} c_w} \tag{1}$$

Cooling tower efficiency (η). The cooling tower efficiency is the ratio of actual cooling (range) to the theoretically possible maximum cooling (that is, when the approach is zero), as shown in Equation (2):

$$\eta = \frac{t_{in} - t_{out}}{t_{in} - T_{w,in}} \times 100 \tag{2}$$

Theoretically, an approach of zero means the tower is 100% efficient. Industrial cooling towers typically have an approach temperature between 4° and 8.5°C, and an efficiency between 70 and 75% [2]; in this case, the efficiency is 75%.

Evaporation loss and air needs

Method 1. The evaporation loss and air flow requirement through the tower can be evaluated by solving the mass and energy balance equations simultaneously.

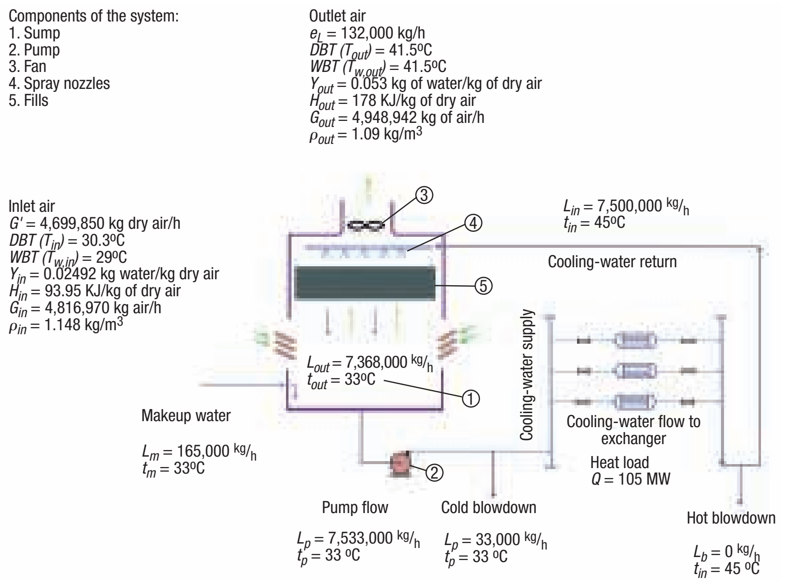


FIGURE 2. This schematic diagram depicts the parameters of the case study cooling tower system. Note: All three cells and three fans are lumped together and shown as a single unit

Use Equation (3) to apply a mass balance for the entire section of tower. As shown in Equation (3), the amount of water evaporated (e_L) in the down-pouring liquid is the difference between the inlet liquid flow (L_{in}) and the sum of the outlet liquid flow (L_{out}) and the drift loss (d_L). It is equal to the difference of moisture content of air across the tower.

$$e_L = L_{in} - L_{out} - d_L = G'(Y_{out} - Y_{in}) \tag{3}$$

Where:
G' = the quantity of dry air flow (which remains the same at the inlet and outlet air streams), kg of dry air
Y = absolute humidity, kg water/kg dry air/h
The subscripts *in* and *out* refer to the entry and exit locations.

The overall energy balance is given by Equation (4):

$$L_{in} h_{in} + G' H_{in} = L_{out} h_{out} + G' H_{out} + d_L h_d \tag{4}$$

Where:
h = the liquid enthalpy, kJ/kg water
H = the moist air enthalpy, kJ/kg dry air
Substituting L_{out} from Equation (3) into Equation (4), and assuming the enthalpy of the drift water h_d is h_{out}, and simplifying Equation (4), one gets Equation (5):

$$L_{in} c_w (t_{in} - t_{out}) + e_L c_w t_{out} = G'(H_{out} - H_{in}) \tag{5}$$

Solving both mass and heat balance equations [Equations (3) and (5)] simultaneously, the evaporation

NOMENCLATURE

c_w = Specific heat of water, kJ/kgK
d_L = Drift loss, kg/h
e_L = Evaporation loss, kg/h
G = Air flowrate (wet), kg of air/h
G' = Air flowrate (dry), kg of dry air/h
h = Water enthalpy, kJ/kg
H = Moist air enthalpy, kJ/kg
L = Liquid flowrate, kg/h
O_L = Other losses (seal leak, pipe leaking, and so on) in the system, kg/h
Q = Heat load, kW
t = Water temperature, °C
T = Air temperature, °C
Y = Air humidity, kg water/kg of dry air
ρ = Moist air density, kg/m^3
λ_o = Latent heat of vaporization of water, kJ/kg
η = Cooling tower efficiency, %
ΔT = Range, °C
Subscripts
d = Drift water
b = Blowdown
in = Inlet location
m = Makeup water
out = Outlet location
p = Pump
w = Wet-bulb temperature

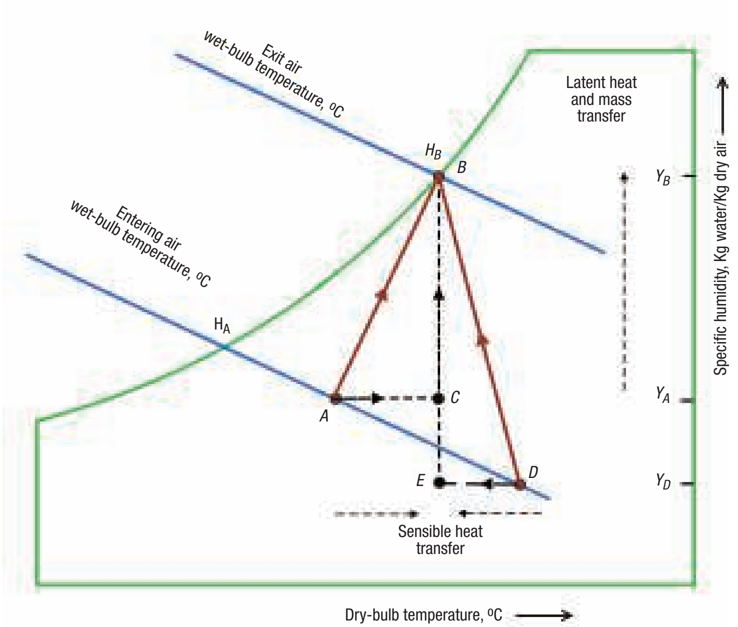


FIGURE 3. In this psychrometric chart, the relevant process condition from the case history is marked as vector AB

loss (e_L) and the dry-air requirement (G') are estimated as 132,000 kg/h and G' = 4,699,850 kg dry air/h, respectively.

Method 2. Depending on the temperature of the inlet air (whether it is hot or cold), the air can be either heated or cooled as it travels along the height of a cooling tower. In the psychrometric chart shown inFigure 3, the entering condition of the air is denoted by point *A*, and the exit air (which is completely saturated with water) is denoted by point *B*. The enthalpy difference of dry air is (H_A–H_B). The vector *AB* is the sum of the two components. The horizontal component *AC* represents the sensible heating of air, and the vertical component *CB* is the latent heating of air. In a cooling tower, it is also possible to cool the air if the inlet air condition is at *D* [*3*]. At point *D*, the air is hot and dry, when compared to the air at point *A*.

The component *DE* is the sensible air cooling, and the component *EB* is the latent heating of air. The net heat received by the air is the difference between the latent air heating and the sensible air cooling.

In the case of the *AB* process, the dry-bulb temperature of the air is increased at the exit — that is, the exit air becomes hot compared to the inlet air. But in the case of *DB*, the dry-bulb temperature of air is decreased and thus the air is cooled at the exit. In both cases, the wet-bulb temperature of the exit air will always be increased compared with that of the inlet air. So, the water flowing through the cooling tower can be cooled by unsaturated air, irrespective of whether the air is hot or cold.

In this case study, from the field measurements of *DBT* and *WBT*, the psychrometric properties, such as absolute humidity, saturation humidity and moist air enthalpy for the inlet air and the outlet air, could be evaluated. The inlet air is marked as point *A,* and the outlet air is marked as point *B* in the psychrometric chart. Another hypothetical point *C* is marked in such a way that it has a dry-bulb temperature similar to point *B* and absolute humidity similar to point *A*. It must be noted that the point *C* is a hypothetical and does not correspond to any location in the cooling tower; the point *C* is marked on the chart to see the horizontal and vertical component of vector *AB*. Moist air enthalpy for point *C* is calculated.

The total heat gained by the air (H_B–H_A) has two components: the latent heat transfer (H_B–H_C), and the sensible heat transfer (H_C–H_A). The ratio of latent heat transferred ($e_L\lambda_o$) to the total heat released from the water side is shown by on the left in the Equation (6), and this expression is numerically equal to the ratio of latent heat added to the dry air to the total heat gained by the air, which is shown on the right side in the Equation (6):

$$\frac{e_L\lambda_o}{L_{in}C_w(t_{in}-t_{out})+e_L C_w t_{out}} = \frac{H_B-H_C}{H_B-H_A} \tag{6}$$

From Equation (6), e_L is calculated as 132,000 kg/h. It is to be noted that in this method, the dry air flow (G') is not required. Once e_L is evaluated, G' is estimated from the mass balance equation [(Equation (3)]. The split of latent heat transfer and sensible air heating in this case is about 85% and 15%, respectively.

Makeup water and blowdown

Makeup water (L_m) is added to the sump to compensate for the water losses in the circuit. The water losses include evaporation loss (e_L), drift loss (d_L), blowdown (L_b), and other leakage losses (O_L) in the system, such as losses from the pump seal, piping leak, washdown water and filter backwash.

$$L_m = L_b + e_L + d_L + O_L \tag{7}$$

Drift loss. Small droplets that are entrained by the upward-flowing air stream are collected in a mist eliminator, where they accumulate to form larger drops that are eventually returned to the fill. In general, very little water in the form of droplets is carried along with the air, but those droplets do results in water loss, called drift loss or windage loss. This drift water typically contains dissolved solids and may cause stain, corrosion or damage to nearby buildings and structures. Drift loss is usually about 0.1–0.3% of the circulation water rate (L_{in}).

To compensate for the evaporation loss and drift loss, additional makeup water is added. Since the makeup water typically contains dissolved solids, these solids are typically left behind in the sump water as the water evaporates in the cool-

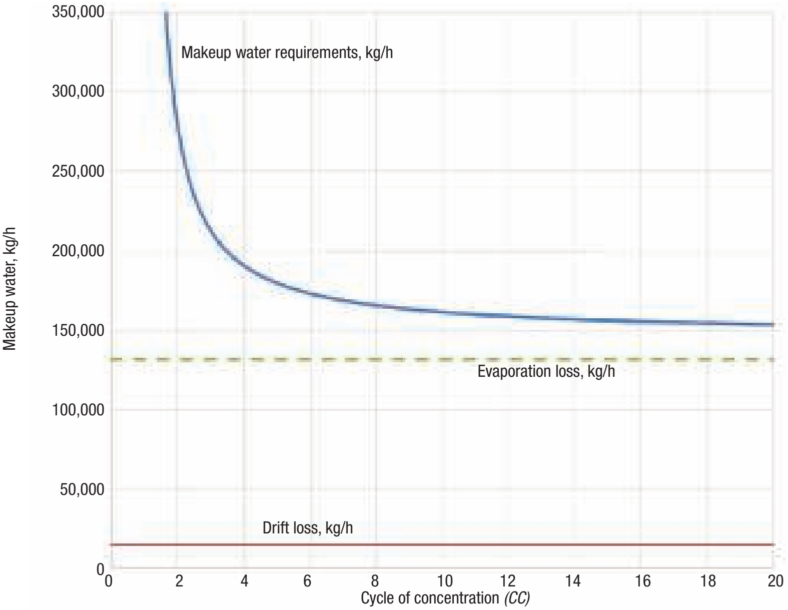


FIGURE 4. Makeup water requirements versus the cycle of concentration are shown here

ing tower. Meanwhile, since the cooling water is a very effective air scrubber, dust and debris present in the up-flowing air is washed out by down-pouring water and collects in the sump. As solids accumulate in the sump, they increase the potential for scale corrosion and biological fouling in the cooling-water circuit. By taking small amounts of water continuously from the cooling tower circuit (blowdown), the concentration of dissolved solids in the cooling water can be reduced below the upper limit of the acceptable range, in order to meet the cooling-water quality specification of the plant.

Blowdown. There are two ways to remove the blowdown — as hot blowdown and cold blowdown (Figure 2). Hot blowdown refers to the continuous removal of water in the cooling-water-return line to the effluent. Since the water is hot at this location, hot blowdown may not be acceptable in some applications due to potential environmental impact; in other cases, it is desired, since it reduces throughput to the cooling tower and increases overall cooling performance.

Cold blowdown refers to the continuous removal of water from the cooling-water pump outlet to the effluent [*4*]. Drift loss and any leakage loss from the system are also considered as blowdown, since these streams contain dissolved solids (but such losses are unintentional).

The amount of water blowdown is established by calculating the cycle of concentration (*CC*), which is defined as the ratio between the amount of solids dissolved (mostly chlorides) in the blowdown and in the makeup water, using Equation (8):

$$CC = \frac{C_b}{C_m} = \frac{L_m}{L_b} \tag{8}$$

Assuming drift loss and leakage losses are negligible, and solving the water-balance shown below in Equation (9):

$$L_m = L_b + e_L \tag{9}$$

The dissolved-solids balance shown below in Equation (10), the blowdown is calculated using Equation (11):

$$L_m c_m = L_b c_b + e_L(O) \tag{10}$$

$$L_b = e_L\left[\frac{1}{CC-1}\right] \tag{11}$$

Further, the amount of makeup water needed is estimated, including drift, using Equation (12):

$$L_m = e_L\left[\frac{1}{CC-1}\right] + e_L \tag{12}$$

The required makeup water mainly depends on evaporation loss and the *CC* calculated above. It is to be noted from Equation (11) that the minimum value of *CC* to be considered is 2, which requires blowdown to be at the same amount of water as the amount lost in evaporation. Any attempt to reduce the *CC* below 2 results in a significant amount of makeup water, as shown in Figure 4.

Higher *CC* means that C_m tends to zero (indicating good quality of the makeup water). But, this is achieved at the cost of water treatment of the source water. A typical cycle of concentration (*CC* = 5) is considered in this case study for the optimum requirement. Based on the evaporation loss and cycle of concentration, cold blowdown and makeup water are calculated using Equation (11) and Equation (12), as 33,000 kg/h, and 165,000 kg/h, respectively. Further, assuming 0.2% drift loss and no system leak, makeup water needs to be considered as 180,000 kg/h. ■

Edited by Suzanne Shelley

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Solids Processing

special advertising section

CHEMICAL ENGINEERING
ESSENTIALS FOR CPI PROFESSIONALS
Access Intelligence

Size reduction, packaging, conveying, drying, sampling, pastillation, level measurement, mixing, weighing, and troubleshooting feature in this month's solids handling showcase


IMAGE: DAVID STANLEY / FLICKR

Wood chips, shown here, are just one of the thousands of granular materials produced or consumed by the chemical process industries

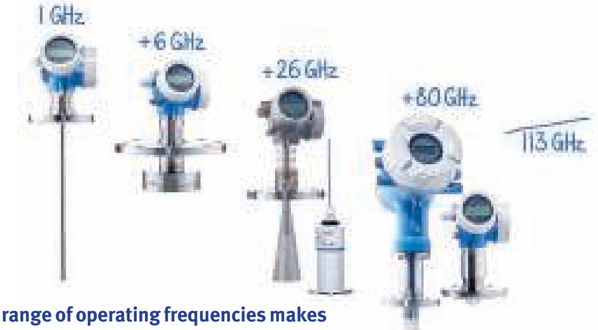
Handling and processing solid materials in the form of granules, powders, pellets and pastilles has always been a key part of the chemical engineering toolkit. This special advertising section highlights just a few of the many ways particulates are transferred, processed, and packaged.

On the following pages you will find a twin-shaft mixer featuring a central fluidized zone; fluidized-bed dryers; dispersers for mixing powders efficiently into liquids, and venturis for removing particulates from gas streams; powder sampling systems; a jet mill for ultrafine powders; systems to turn viscous melts into free-flowing pastilles; and radar level measurement devices with long range and high accuracy. Alongside these dedicated operations are also a number of companies offering a broad range of expertise in conveying, weighing, packing, and palletizing solid materials. ■

Inside:

Get on the right wavelength

Endress+Hauser's level measurement range offers the right answer for each application



A full range of operating frequencies makes it easy to choose the best one for each application

Endress+Hauser has completed its radar level measurement portfolio with the new Micropilot FMR60, FMR62 and FMR67. The Micropilots are the first 80 GHz instruments to follow the safety-by-design concept and to be developed according to IEC 61508. They are also the first level instruments to support Heartbeat Technology, as already used in many of the company's flowmeters.

At the beginning of 2016 Endress+Hauser launched the Micropilot NMR81, the first high-accuracy instrument based on 80 GHz technology for tank gauging in chemical, oil and gas applications. Now the company has introduced the Micropilot FMR6x models, which promote safety and simplicity alongside high accuracy.

For the first time, an 80 GHz process radar is available that follows the safety-by-design philosophy, thus making customers' lives easier in terms of safety. The new products also offer a wide selection of Ex approvals.

Improved focusing of the radar signal as well as dynamic processing algorithms provide reliable, stable measurements with a range of up to 125 m and an increased measuring accuracy of ±1 mm. Measurements are unaffected by baffles or other obstacles on the tank walls. Thanks to the innovative antenna design, the system also ignores product buildup and condensation. Interactive software makes commissioning fast and easy.

Instruments with their own "pulse" – the so-called Heartbeat Technology – have so far appeared mainly in Endress+Hauser's flow measurement range. The addition of Heartbeat to the new radar gauges allows documented verification of every measured value, as well as facilitating trend recognition by collecting data for predictive maintenance. Automatic test protocols and a guided SIL proof test ensure that each instrument meets standards for safety and accuracy at all times.

Endress+Hauser uses the slogan "113 GHz + Your Wavelength" for its radar technology to emphasize how individual customers' needs are best met by a full range of frequencies (see image, above). 1 GHz, for example, is used for guided radar in applications involving foam and liquids of low dielectric constant. 6 GHz is best for turbulent liquids and heavy condensation. 26 GHz provides tight focusing and is suitable for 90% of applications, even those involving turbulence. 80 GHz provides the best focus (a 3-deg. beam angle), a measuring range of up to 125 m, and the highest accuracy.

In the end, radar is not the best solution for every application. Endress+Hauser therefore offers a complete portfolio of level measurement technologies. **www.yourlevelexperts.com/en**

Ultra-high speed powder dispersion made simple

Ross SLIM Technology employs high shear for rapid and complete mixing of powders into liquids, avoiding agglomerates and dust formation

The **Ross** Solids/Liquid Injection Manifold (SLIM) is a technology for dispersing challenging powders like fumed silica, gums, thickeners and pigments using a specially modified high shear rotor/stator generator.

In both batch and inline designs, the SLIM is easy to retrofit into almost any process. In an inline set-up, the SLIM mixer pumps liquid from the recirculation tank while simultaneously drawing powders from a hopper. As the liquid stream enters the rotor/stator assembly, it immediately encounters the powder injection at the high shear zone. The mixture is then expelled through the stator at high velocity and recirculated back into the tank. In just a few short turnovers, solids are completely dissolved or reduced to the desired particle size.

This method for high-speed powder injection is ideal for dispersing small concentrations of hard-to-wet solids like CMC or xanthan gum (>5%). It is equally effective for solid loadings as high as 70%, as in the case of titanium dioxide or magnesium hydroxide slurries. By introducing solids sub-surface where they are instantly subjected to vigorous agitation, issues like floating powders, excessive dusting and formation of stubborn agglomerates ("fish eyes") are eliminated. Because the SLIM generates its own vacuum for powder induction and does not rely on external eductors or pumps, it is free of clogging and simple to operate.

Several models are available including automated skid packages where the SLIM mixer is piped to a jacketed tank and supplied with flowmeters, load cells, solenoid valves, level sensors and thermocouples all integrated into a PLC Recipe Control Panel. Each ingredient addition and process step can be pre-programmed so that mixer speed, mixing time, temperature, composition and batch weight are accurately replicated in every run.


Ross Inline SLIM powder induction mixer with built-in control panel mounted on a portable cart with work bench

Established in 1842, Ross is one of the world's oldest and largest manufacturers of process equipment, specializing in mixing, blending, drying and dispersion. **www.highshearmixers.com**

Handle virtually any bulk solid material

Flexicon stand-alone equipment and automated plant-wide systems convey, discharge, condition, fill, dump and weigh batch bulk materials dust-free

Flexicon engineers and manufactures a broad range of equipment that handles virtually any bulk material, from large pellets to sub-micron powders, including free-flowing and non-free-flowing products that pack, cake, plug, smear, fluidize, or separate.

Individual bulk handling equipment includes: flexible screw conveyors, tubular cable conveyors, pneumatic conveying systems, bulk bag dischargers, bulk bag conditioners, bulk bag fillers, bag dump stations, drum/box/container dumpers, and weigh batching/blending systems. Each of these product groups encompasses a broad range of models that can be custom engineered for specialized applications, and integrated with new or existing upstream and downstream processes and storage vessels.

All equipment is available to food, dairy, pharmaceutical and industrial standards.

For large-scale bulk handling projects, Flexicon's separate Project Engineering Division provides dedicated Project Managers and engineering teams on four continents to handle projects from concept to completion. Working with each customer's preferred engineering firm or directly with their in-house team, Flexicon adheres strictly to the customer's unique standards, documentation requirements and timelines through a single point of contact, eliminating the risk of coordinating multiple suppliers.

Flexicon's worldwide testing facilities simulate full-size customer equipment and systems, verify performance prior to fabrication, demonstrate newly constructed equipment for visiting customers, and study the performance of new designs.


Flexicon offers stand-alone bulk handling equipment as well as plant-wide systems integrated with new or existing processes

In 2015 the company doubled the size of its manufacturing facility and world headquarters in Bethlehem, PA, and also operates manufacturing facilities in Kent, United Kingdom; QLD, Australia; and Port Elizabeth, South Africa. **www.flexicon.com**

Fluidized-bed drying, cooling and granulation

GEA is an expert in the design and manufacture of fluidized-bed dryers, coolers and granulators, including versions for high temperatures and large throughputs

Fluidized-bed dryers, coolers and granulators are used for the controlled removal of residual moisture from powder, crystalline and granular products. Fluidized-bed processing is characterized by gentle drying, cooling or granulation over extended product residence times. The gentle but intimate contact between the drying/cooling gas and the product ensures minimal attrition and dust creation.

GEA continuously develops its fluidized-bed drying technologies to meet the requirements for optimum design across an expanding range of applications. This development, which has been going on for many years, has reached its current high standard based on feedback from more than 1,000 industrial fluidized-bed installations around the globe and more than 30,000 pilot tests carried out in GEA's drying test center.

Attention to airflow and thermal expansion lets GEA create fluidized beds up to 100 m^2

The choice of the optimum GEA fluidized-bed technology depends on the flowrate and properties of the raw material and the utilities available. For large-scale drying, such as in the agrochemicals and minerals industries, GEA has developed a high-temperature fluidizer design giving optimum energy efficiency. The special mechanical design allows free thermal expansion for each dryer module, enabling GEA to offer high-temperature fluidizers with bed areas exceeding 100 m^2.

A unique mechanical flex design ensures minimum thermal and mechanical stress in the construction. The use of multiple drying zones guarantees optimum drying conditions, while a tailored feed inlet spreader design ensures trouble-free back-mixing of moist product in the inlet section. The fluidizing plate itself, which is of a special patented design that GEA refers to as the Gill Plate, is tailor-made for each application to ensure efficient movement of solid material. Combined with the underflow discharge design of the dryer, this ensures that the fluidized bed is continuously self-emptying during operation.

GEA specializes in supplying tailor-made industrial drying systems. The GEA comprehensive product range for industrial dryers includes fluidized-bed dryers, spray dryers, flash dryers, ring dryers, rotary dryers, spray congealing plants, and spray drying absorption plants. **www.gea.com**

Securely bagged and wrapped

BEUMER supplies complete packaging lines for the chemical industry: from bag filling, through palletizing, to shrink wrapping of finished pallets

The BEUMER fillpac FFS: High throughput, availability and a compact design are key features of the system

The **BEUMER Group** supplies complete packaging lines which fulfill the complex requirements of manufacturers in the chemical industry. These include highly efficient filling, palletizing and packaging systems. Customers also get comprehensive support.

When the BEUMER Group implements a packaging line in the chemical industry, here is how it typically works: The bulk material passes from the silo along a conveyor section to the BEUMER fillpac FFS (form fill seal) system (photo, left). This bagging machine is fitted with an integral, high-precision weigher that ensures the accuracy of the bag weights. High-performance palletizers from the BEUMER paletpac range then stack the bags on pallets quickly and accurately. Depending on the product requirements, the palletizers are fitted with rotary-clamp or double-belt turning devices to move the filled bags into the required position. Even bags filled with granulate can be palletized gently, remaining dimensionally stable when stacked.

Finally, customers use high-performance packaging systems in the BEUMER stretch hood series (photo, right) to secure the load. For businesses in the chemical industry, efficient packaging of palletized goods is a crucial competitive factor. The film fits snugly over each product on the pallet, ensuring safe transport and good load stability. The film is highly stretchable and secures the material as it contracts. It is also very transparent and permits a clear view of the packaged goods, including barcode scanning. If a cover film is additionally placed over the top of the pallet, the goods are protected from all six sides.

Many companies in the chemical industry are using the stretch hood process to reduce transport damage and meet more exacting logistical demands

Customers get a complete solution from a single source and have a reliable point of contact for the whole job, BEUMER notes. **www.beumergroup.com**

Repeatable sampling for bulk solids

Sentry Equipment's PR sampler automatically takes samples of free-flowing products from pneumatic conveying lines

The PR sampler is a flexible quality control solution that is ideal for products such as polymer pellets

Designed to eliminate the degradation of product, the **Sentry** Model PR takes point samples of free-flowing material from dilute- and dense-phase pneumatic conveying lines. The PR takes a sample when a solenoid-controlled air cylinder operates a reciprocating probe into the product flow. This sampler is suitable for pellets, powders, flakes and granules.

The PR sampler features a retractable probe design that isolates process pressure from the sample container. The sample tube and auger capture a fixed sample volume. The sample probe retracts from the line to avoid any restriction in the product flow between sample cycles. It enables rapid cycling to acquire a large volume of sample over a short period of time. A standard purge port assures full sample discharge. The PR features rugged stainless steel construction with optional sample tube materials for abrasive or non-free-flowing products. Its controllers and remote operating modules are available in a variety of configurations.

The ability to get a sample whenever desired, in the volume desired, in the same manner every time is a benefit for control efforts to accurately diagnose process performance. Automated sampling removes the unknown of whether a sample was taken properly and at the time required.

For example, when polyethylene pellets are manufactured, each batch is sampled a number of times for quality control, with the first sample needed to continue running the process. In this application, a Sentry PR sampler is mounted in the conveying lines before they exit the building to loadout.

The sampler's automation is controlled by a Sentry P2B-4/7 sampler controller. Using the sampler controller, an operator can gather the first required sample by pressing the "hand" option as many times as required to collect the needed sample volume. The operator then changes the container, places the sampler controller in "auto", and utilizes the "batch" option. This allows the sampler to collect samples automatically at a preset rate.

Using the batch option on the sampler controller, a facility can continuously collect composite samples representative of the process over the course of time. This reduces waste as well as the possibility of overfilling the sample container, which could result in wasted product or a spill.

By implementing a Sentry PR automatic sampling solution within their processes, plastics manufacturers can improve efficiency and gain confidence that their sample analysis results are based on truly representative product samples.

www.sentry-equip.com

Venturi scrubbers remove particulates reliably

Venturi scrubbers from CR Clean Air offer a robust way to remove particulates and acid gases from gas streams, with high-energy versions able to tackle particles below 1 µm

High-energy venturi scrubbers like this one, shown complete with separator tank, compare favorably with wet electrostatic precipitators on sub-micron particulates

CR Clean Air offers a wide range of jet venturis and high-energy venturis to remove particulate materials from gas streams. The company's jet venturis remove any particles larger than 1 µm, plus acid gases such as HCl and SO_2. The venturis themselves are often sold complete with vapor/liquid separator tanks. Since large liquid-to-gas ratios are needed to maximize performance, the scrubbing liquid is generally recirculated from the tank back to the venturi. The jet induces a small increase in gas differential pressure, eliminating the need for an exhaust fan to pull it through the scrubbing system.

For smaller particulates, the company's high-energy venturis can be used (photo, right). These units impart a large pressure drop (as much as 60 in. w.c.) on the gas in order to force the particulate into the scrubbing liquid. Both fixed- and variable-throat designs are available, and removal rates as high as 99.9% can be realized. High-energy venturis are the technology of choice for wet removal of submicron particulates, and compare favorably to the cost of a comparable wet electrostatic precipitator (WESP).

CR Clean Air offers its high-energy units complete with cyclonic separators. The company can also provide full systems, including an exhaust fan and liquid recirculation pumps, skid-mounted with valves, instruments and controls. Both metallic and FRP constructions are available, and each system is customized around the requirements of the process.

The company has over 50 years of experience spanning a wide range of industries, from chemicals to pharmaceuticals, and semiconductors to refineries, with thousands of installations worldwide. So whether you need a jet venturi, high-energy venturi or even a packed tower, CR Clean Air has the solution to your emission control challenge.

www.crcleanair.com

Creating free-flowing powder to boost energy storage

The Micronizer jet mill from Sturtevant relies on particle-on-particle attrition to cause particle size reduction that boosts power capabilities within batteries

To create powerful batteries, energy storage manufacturers need to increase the surface area of the lithium- or carbon-based materials that act as electrolytes between the positive and negative electrodes. During size reduction, it is critical that the lithium or carbon not become contaminated and maintain its purity for the final product. The **Sturtevant** Micronizer brand jet mill reduces the particle size of lithium and carbon powders without contamination or heat buildup.

The Micronizer employs high-pressure compressed air, steam, or other gas to disperse and deagglomerate the particles. It consistently produces low-micron-sized particles whose chemical reactivity is increased compared to larger particles thanks to their far greater surface area.

The Micronizer's open manifold design allows complete access to the internal material grinding chamber and compressed air chamber for easy cleaning, product changeover, or inspection. There are no dead zones to trap material, no moving parts, and no grinding media or lubricants to contaminate the lithium or carbon being milled.

The Micronizer utilizes a unique fluid energy grinding system to generate particle-on-particle impact without raising temperatures that could damage the material being milled. Its high-performance design surpasses the economical fineness limit of mechanical grinders.

Sturtevant can solve the most challenging wear and contamination problems using interchangeable wear-resistant liners such as PureLine, ShieldOx, and ArmorLine, and specifically engineered coatings like Lubriguard to provide contamination-free wear protection and enhance product quality and throughput.

Sturtevant offers a fully equipped test facility for conducting customer trials with the objective of determining the optimum equipment and system layout for each application. Sturtevant invites customers to witness testing while determining the best way to achieve fine particle sizes and understand grinding characteristics.

www.sturtevantinc.com

The Sturtevant Micronizer jet mill yields particle sizes down to 0.25 µm. Its use of fluid energy allows it to out-perform mechanical grinding systems without risk of contamination.

Fast, homogenous mixing

The Bella XN fluidized zone mixer from Dynamic Air is a twin-shaft design that uses a "weightless" central fluidized area to provide thorough yet gentle mixing of dry products

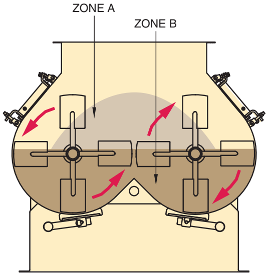
The twin-shaft Bella mixer

The Bella fluidized-zone twin-shaft paddle mixer by **Dynamic Air** achieves fast, high-capacity, low-shear, precision mixing of either dry bulk solids or liquids with solids. Regardless of particle size, shape or density, materials are mixed with a fast, efficient, and gentle action, with typical mixing times of 15–30 s. A weightless zone created by low-speed counter-rotating paddles generates low friction without shear. This makes it ideal for abrasive products and fragile products that cannot tolerate rough handling. Even flakes or spray-dried bodies remain intact.

The Bella mixer consists of twin drums which have two counter-rotating agitators with specifically angled paddles. The paddles sweep the entire bottom of both mixer drums and yet allow the mixer to be started under full load (Figure 1). The material in the mixer moves in a horizontal counter-clockwise direction at the perimeter while simultaneously moving both left and right in the center (Figure 2). The material in Zone B (Figure 1) is in its normal gravimetric state as it is being moved and dispersed. In Zone A, a weightless zone is created which effectively lifts the ingredients to an almost weightless state, allowing them to move freely and randomly, regardless of particle size and density. Thus the two zones' interaction becomes highly efficient as every particle moves rapidly to create a highly homogeneous mix, the key to the Bella mixer mixing technology for fast, precise mixing.

The Bella mixer is available in stainless steel and mild steel construction.

www.dynamicair.com/products/mixers.html



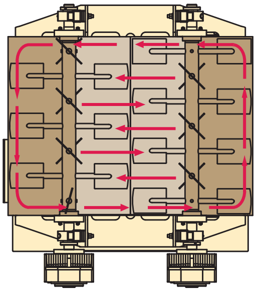


Figure 1 (right, top): In Zone A, fluidization promotes thorough mixing. Figure 2 (right): Material interchange between the two drums

A wide range of custom material handling equipment

Material Transfer is a premier manufacturer of custom material handling equipment, with products including bag conditioners, dischargers, and filling systems

Material Transfer's Bulk Bag Conditioners ensure that products are free-flowing

Material Transfer is an industry leader in the custom design and manufacture of material handling equipment and systems for dry powders and bulk solids. A unique combination of application-focused engineering, award-winning designs and exclusive features results in equipment that offers class-leading quality, value, durability, ease of use, and performance.

Equipment is fully assembled, inspected, and factory tested prior to shipment to ensure reliable performance and customer satisfaction, the company says.

Material Transfer's application experience includes pharmaceuticals, food, chemicals, electronics, aggregate, agriculture, foundry, manufacturing, mining, packaging, pet food, petroleum, plastics, plating, stamping, governmental, explosives and automotive. Experience in dust-tight handling of hazardous materials in hazardous environments, with explosion-proof or intrinsically safe equipment, has placed Material Transfer in the forefront of designing and building custom material handling equipment for these applications.

Over 95% of the equipment Material Transfer manufactures is custom-designed for a customer's particular application requirements. Material Transfer has a team of talented engineers with the latest 3-D software for equipment design, professional metal fabricators and machinists with the latest fabrication and CNC machining technologies, and an experienced team of machine assemblers to build its products.

Material Transfer's product line includes:

- Material Master Bulk Bag Conditioners to quickly and safely return hardened materials to a free-flowing state (photo);
- Material Master Bulk Bag Dischargers with patented technology to provide clean, dust-tight discharge;
- Material Master Bulk Bag Fillers – from economical 4-post units to fully automated, high-output PowerFill filling systems.
- Container and drum dischargers to empty containers of any size at heights to 40 ft., with dust-tight Lift & Seal system or open discharge, and patented system that allows 180° rotation.
- Integrated systems to meet customers' requirements.

www.materialtransfer.com

Engineered solutions for reliable bulk solids flow

With more than half a century of experience, Jenike and Johanson relies on a strong technical team to cure – or preferably forestall – clients' problems with powder flow

Skilled technical staff have always been key to Jenike & Johanson's success

Jenike & Johanson, Inc. is the world's leading technology company for bulk material handling, processing, and storage. The firm delivers engineered solutions to achieve reliable powder and bulk solids flow, based on proven theories and decades of project experience. With a skilled, highly technical team of experts and industry-leading innovations, Jenike & Johanson has successfully delivered bulk material engineering solutions for more than 55 years.

Bulk materials and their flow properties are at the core of all Jenike & Johanson's work. Every project (7,500+ to date) is truly unique, and clients are offered maximum flexibility in selecting services to meet their bulk material handling needs. Decisions made during the feasibility and engineering stages of a project are critically important for success. If bulk solids systems are not engineered from the outset to handle the unique characteristics of the materials, process start-up can be significantly delayed and design capacity may never be reached.

The engineers at Jenike & Johanson are renowned experts in bulk material engineering. They are frequent keynote speakers at industry events, routinely deliver webinars and customized courses, and publish technical articles in top industry journals.

75% of all chemicals are handled in bulk solid form during manufacturing. When feeding powders to reactors or conveying wet cake from a centrifuge to a dryer, poor material flow can result in limited throughput, non-uniform product, dust emissions, or spillage. Reliable solids handling at chemicals plants will improve operations involving blending, extrusion, pneumatic or mechanical conveying, and heating or cooling. Reliable powder flow and a first-in, first-out vessel discharge pattern (mass flow) minimizes cross-contamination between batches and maximizes process efficiency.

Jenike & Johanson offers a wide range of services to effectively support any engineering project in the chemical industry:

- on-site audit to review problems;
- raw, intermediate, final grade flow testing;
- storage vessel, chute, and feeder design;
- pneumatic conveying testing;
- silo structural engineering (new, retrofit);
- pilot-scale process modeling.

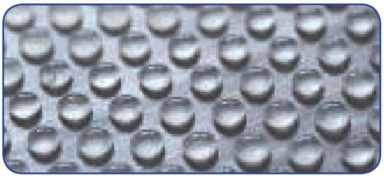
Whether conveying raw materials from storage silos to a process, feeding powders to a reactor, or packaging products in bulk, Jenike & Johanson can help achieve reliable material flow and high throughput.

www.jenike.com

Continuous cleanroom pastillation

Sandvik's Rotoform pastillation system is available in GMP versions for pharma, cosmetic and food applications

Achieving a successful pharmaceutical audit for cleanroom pastillation requires a GMP-compliant production unit with professional documentation. The **Sandvik** Rotoform granulation system meets this requirement and converts liquid melts into solid pastilles.



Uniform pastillation thanks to Rotoform

Every aspect of the Rotoform system has been designed to ensure GMP compliance. All piping connections are quick-release types for easy cleaning, and the Rotoform is powered by a maintenance-free drive belt.

Rotoform uses a heated cylindrical stator supplied with liquid product, and a perforated rotating shell that turns concentrically around the stator. Product droplets are deposited onto a stainless steel belt.

Indirect cooling via the endless steel belt eliminates any risk of cross-contamination between product and cooling water, while a hot-water-based belt cleaning system eliminates the risk of cross-contamination between batches.

All working parts that can come into contact with the product are stainless steel for easy cleaning and maximum hygiene. The structural materials have a very low surface roughness. All components (i.e. gaskets) inside the Rotoform are made from materials compatible with the respective product.

The whole machine is covered with hoods, with an extractor fan to avoid pollution of ambient air. The control system is supported by cameras that transmit images to an outside terminal.

The Sandvik Rotoform system is widely used across the food, cosmetic and pharmaceutical industries for products such as chocolate, lipstick and suppository mass.

www.processsystems.sandvik.com

Industry events for solids handling

PHOTO: © MESSE DÜSSELDORF/CONSTANZE TILLMANN

Packaging in action at interpack

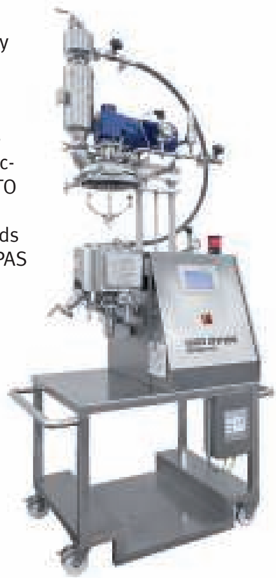
A large number of dedicated exhibitions, conferences and training sessions shows the importance of solids handling and packaging to the chemical process industries. Below is a short calendar of the most important these events in the U.S. and Europe this year:

May 4–10, 2017
interpack 2017
Messe Dusseldorf, Düsseldorf, Germany
May 10–11, 2017
SOLIDS Dortmund
Messe Westfalenhallen Dortmund, Germany
May 16–18, 2017
Toronto Powder & Bulk Solids
Toronto Congress Centre, Ont., Canada
June 19–21, 2017
Eurodrying 2017 – European Drying Conference
University of Liège, Liège, Belgium
September 14, 2017
Bulk Solids Handling Workshop
organized by *Chemical Engineering*
Sky Philadelphia, Pa., U.S.
September 26–28, 2017
POWTECH 2017
Exhibition Centre Nuremberg, Germany
September 27–29, 2017
The 2017 Powder Show & Conference
Greater Columbus Convention Center, Columbus, Ohio, U.S.
October 4–5, 2017
SOLIDS Rotterdam 2017
Ahoy Rotterdam, Rotterdam, Netherlands.

A small footprint takes a big step

EKATO's new lab dryers are flexible and versatile

EKATO SYSTEMS Germany is promoting a range of "desktop-type" laboratory-scale dryers. With working volumes of 3–6 l, the VPT3 dryers cater to a growing demand for drying tests in vertical apparatus, and also allow small batches of product to be manufactured. Scale-up is easy because the design matches EKATO's full-scale dryers.



EKATO VPT3 laboratory dryers handle both free-flowing and pasty materials, and are ideal for process development

The base model in this plug-and-play system is a drying vessel with a water jacket for heating or cooling. The variable-speed motor accepts both the EKATO PARAVISC impeller for free-flowing solids and the EKATO ISOPAS impeller for pasty products.

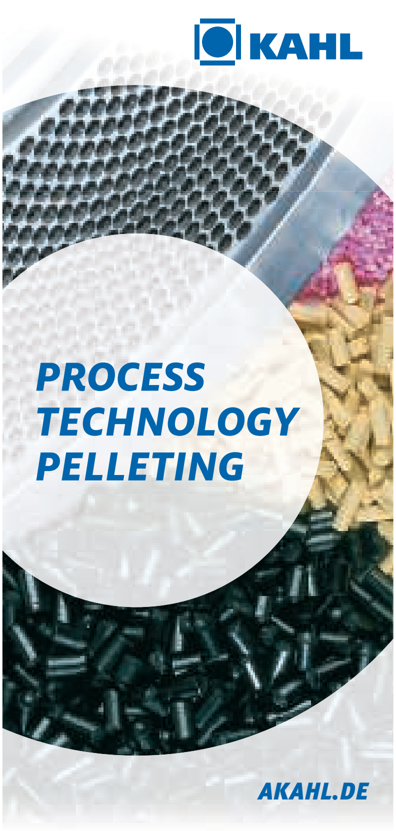
Working pressures range from –1 to +2 bar g. Vacuum is provided by a membrane vacuum pump with condenser and condensate vessel. Working temperature is up to 131°C. Contact parts are made from stainless steel, with other materials available on request.

Control is via a touch screen, with an electric motor used to lift the vessel lid. A data logger and an interface for remote data transfer are available on request.

Just like EKATO's production-scale dryers, the VPT3 dryers can handle thick paste-like products just as easily as low-viscosity suspensions. For shear-sensitive active ingredients that require gentle drying, the EKATO PARAVISC impeller combines uniform product circulation with good heat transfer. Varying the shaft speed allows experimenters to find the operating point that reliably achieves the shortest drying times or the best product properties. A rental unit is available.

www.ekato.com

Product Showcase



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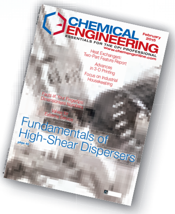

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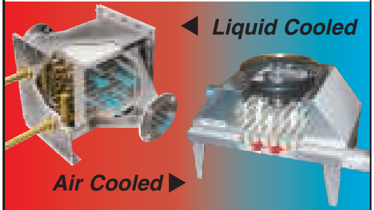

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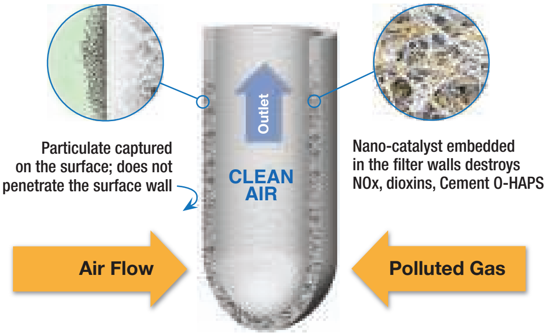
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Economic Indicators

2015 🟪 2016 🟦 2017 🟩

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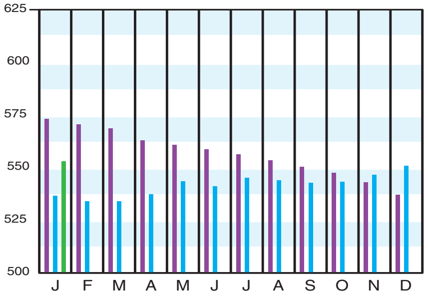
CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Jan. '17 Prelim.	Dec. '16 Final	Jan. '16 Final
CE Index	553.0	550.9	536.4
Equipment	664.1	661.0	640.5
Heat exchangers & tanks	578.3	573.7	551.7
Process machinery	668.6	667.9	649.3
Pipe, valves & fittings	835.2	833.9	795.0
Process instruments	398.5	396.9	378.9
Pumps & compressors	971.3	973.5	979.1
Electrical equipment	512.6	512.1	509.0
Structural supports & misc	722.4	713.9	701.9
Construction labor	324.0	324.4	319.6
Buildings	550.0	547.0	537.7
Engineering & supervision	313.9	313.6	316.6

Starting with the April 2007 Final numbers, several of the data series for labor and compressors have been converted to accommodate series IDs that were discontinued by the U.S. Bureau of Labor Statistics

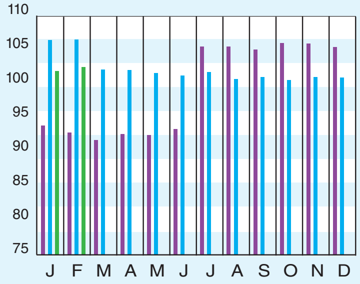
Annual Index:
2009 = 521.9
2010 = 550.8
2011 = 585.7
2012 = 584.6
2013 = 567.3
2014 = 576.1
2015 = 556.8
2016 = 541.7



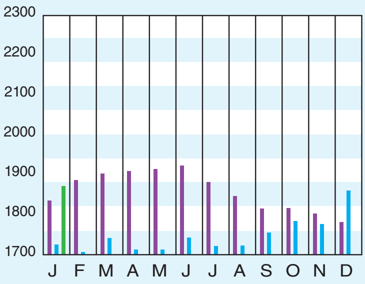
CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS		YEAR AGO
CPI output index (2012 = 100)	Feb. '17 = 102.5	Jan. '17 = 101.9	Dec. '16 = 101.3	Feb. '16 = 101.9
CPI value of output, $ billions	Jan. '17 = 1,872.2	Dec. '16 = 1,861.8	Nov. '16 = 1,785.0	Jan. '16 = 1,672.5
CPI operating rate, %	Feb. '17 = 75.1	Jan. '17 = 74.8	Dec. '16 = 74.4	Feb. '16 = 74.9
Producer prices, industrial chemicals (1982 = 100)	Feb. '17 = 244.3	Jan. '17 = 241.9	Dec. '16 = 239.2	Feb. '16 = 219.2
Industrial Production in Manufacturing (2012=100)*	Feb. '17 = 104.5	Jan. '17 = 104.0	Dec. '16 = 103.5	Feb. '16 = 103.3
Hourly earnings index, chemical & allied products (1992 = 100)	Feb. '17 = 170.1	Jan. '17 = 169.0	Dec. '16 = 170.3	Feb. '16 = 158.6
Productivity index, chemicals & allied products (1992 = 100)	Feb. '17 = 103.0	Jan. '17 = 103.4	Dec. '16 = 103.1	Feb. '16 = 103.1

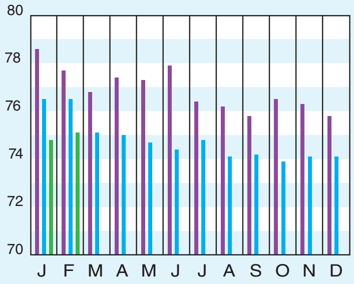
CPI OUTPUT INDEX (2000 = 100)†



CPI OUTPUT VALUE ($ BILLIONS)



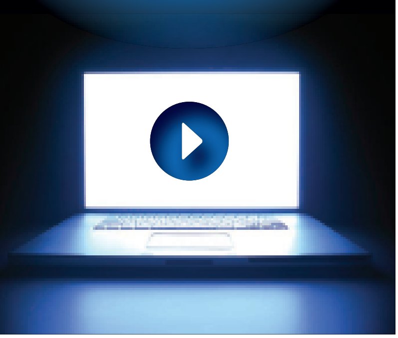
CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
†For the current month's CPI output index values, the base year was changed from 2000 to 2012
Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

The preliminary value for the January *CE* Plant Cost Index (CEPCI; top; the most recent available) represents a gain from the last reading of 2016. With that, the annual CEPCI number for 2016 was calculated to be 541.7, using the average of the monthly values from the year. The annual value is lower than the corresponding value from 2015, and continues a downward trend in annual numbers since the most recent peak in 2011. However, the preliminary monthly CEPCI number for January 2017 stands at 3.1% higher than the corresponding value from January 2016. Meanwhile, the latest Current Business Indicators (CBI; middle) for February 2017 saw an increase in the CPI Output Index and a small decrease in the Productivity Index.

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