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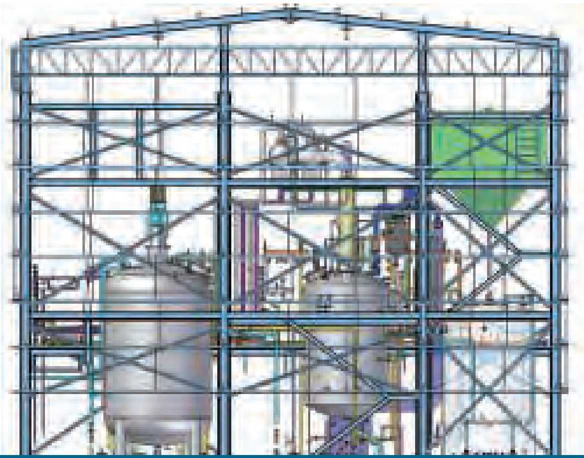

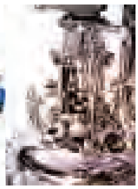

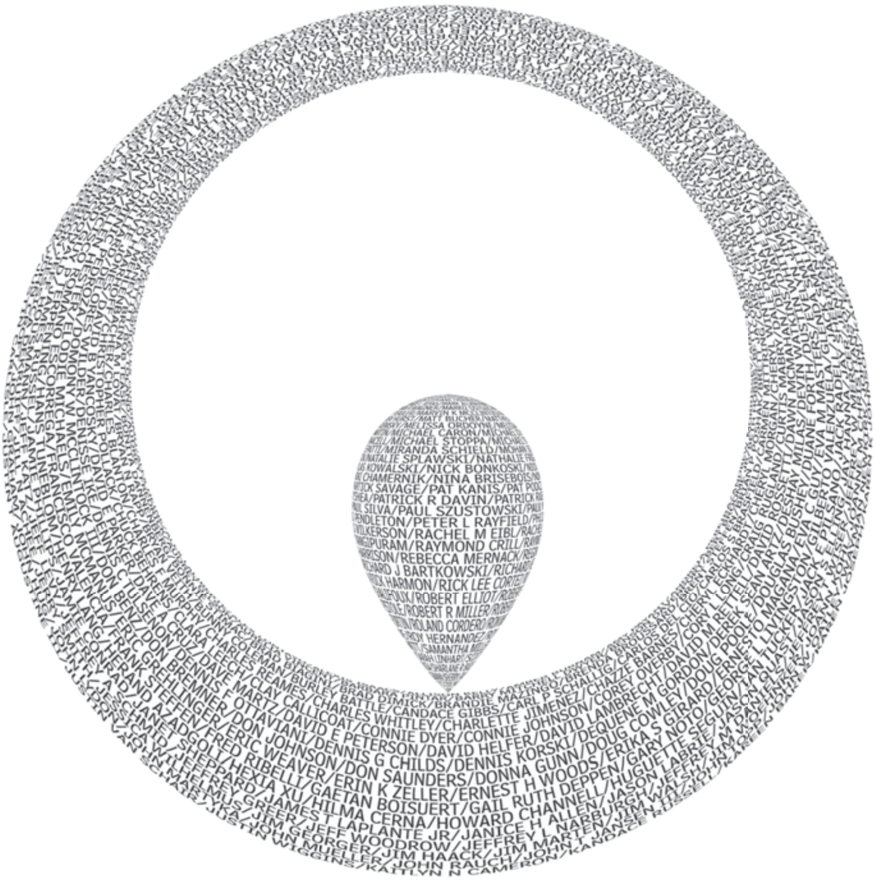

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Editor's Page

Toxicity under the microscope

Last month, two significant cracks were found in the cornerstones of conventional thinking about how chemicals in the environment make their way into the human body and other living organisms, possibly leading to damaging effects. The timing is particularly apropos, as the first registration deadline of November 30 approaches for the EU's regulation on Registration, Evaluation, Authorization and Restriction of Chemical substances (REACH), and the U.S. Congress, under H.R. 5820, considers the first real revamp of the Toxic Substances Control Act (TSCA) since its adoption in 1976.

The issue specifically pertains to bioaccumulation, an important component of the exposure hazard assessment and risk assessment of organic chemicals. In an article published in *Env. Sci. Tech.* (Aug. 11, 2010), Michael S. McLachlan and a team of researchers challenge two of the central paradigms that form the backbone of bioaccumulation assessment in existing regulations. They assert the following: 1) The assessment of chemical bioaccumulation should be done from a multimedia perspective (by referencing the chemical quantity/concentration in an organism to the chemical quantity/concentration in its total environment, not just one medium of that environment, such as water; and 2) The biotransformation rate is a more important determinant of bioaccumulation than partitioning properties.

The first point applies to chemicals in general, McLachlan says. The thinking in the past has been that bioaccumulation should be assessed by comparing the chemical's concentration in an organism to its freely dissolved concentration in the medium in which the organism lives. Under that thinking, the bioaccumulation factor for a fish would be C_{fish}/C_{water} for instance. More recently air has been introduced as the reference medium for calculating bioaccumulation for air-breathing organisms. McLachlan says that this approach leads to an incorrect ranking of the relative bioaccumulation of different chemicals. "It does not take into consideration that many chemicals in the environment are sequestered into media other than the exposure medium. It overvalues the likelihood of such contaminants being transferred from the environment to biota, and hence undervalues the bioaccumulation of chemicals that are primarily found in the exposure medium and that are not sequestered into other media." The distortion can amount to many orders of magnitude, he adds.

Meanwhile, the second point applies to non-ionic substances that are not highly volatile or very water soluble. Currently partitioning properties, in particular K_{OW} (the octanol/water partitioning coefficient), are used in many regulatory frameworks as screening criteria to identify potentially bioaccumulative substances. "We are not aware of any regulations that utilize measures of biotransformation to screen for bioaccumulative chemicals," McLachlan says. "But — for chemicals that aren't highly volatile or water soluble — our work demonstrates that the rate of biotransformation is what will determine whether a chemical is bioaccumulative or not."

McLachlan does not expect a rapid impact. However, he does predict that this work will, in the mid-term, lead to changes in how bioaccumulation is assessed. He notes that there are scientific challenges that have to be overcome before better screening assessments for bioaccumulative chemicals can be adopted in regulations. "We hope that our work will encourage the development of methods to screen for biotransformation, and that when they are ripe these methods will be incorporated into the lower tiers of bioaccumulation assessment frameworks in regulations such as REACH and TSCA." ■

Rebekkah Marshall

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Texas cement plant ready to share award-winning process

On May 5, 2010 Texas Lehigh Cement (Buda, Tex.; www.texaslehigh.com) was awarded an Environmental Excellence Award by the state environmental agency Texas Commission for Environmental Quality (TCEQ; Austin; www.tceq.state.tx.us). The company won for its alternative fuel project because of its outstanding community partnership process. Judges for the award said they could see potential for other plants and other industries to apply this type of process, and Texas Lehigh Cement president Robert Kidnew has said he would be happy to help other firms understand it and use it for themselves.

Alternative fuel voluntary testing protocol. In 2008 Texas Lehigh Cement needed a more economic and reliable source of fuel to supplement the coal used in its kiln. As they started to evaluate a locally manufactured fuel made of chipped waste wood and chipped waste tires, they hired Carbon Shrinks (Austin, Tex.; www.carbonshrinks.com) to develop a community engagement process. Together they designed a voluntary testing protocol that addresses concerns expressed in one-on-one discussions with local environmental critics and community members.

Carbon Shrinks listened to concerns about air emissions and potential odors that are beyond the scope of permits. They designed a process that weighs and scores science-based test results and transparently explains them to a community advisory committee. That process is now available for anyone else to use in their own alternative fuel project.

Economic benefits never more important. The recession has hit all U.S. cement plants hard, so the financial benefit of every project has to be solid. For Texas Lehigh, the return on investment of their voluntary testing protocol stacks up well, and the environmental benefit positions them well for the future. Not only do they now have capacity to use cheaper fuel and the direct experience of using it — showing that the quality of their manufacturing process is uncompromised — they have thoroughly mitigated the risk of expensive community push-back. Potential critics are now friends and allies.

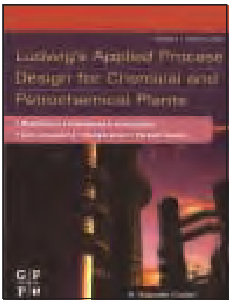
Future market demand for lower-carbon cement. The U.S. Green Building Council and Wal-Mart are both developing supply chain standards that look at the carbon intensity of manufactured inputs. It may only be a matter of time until bid specifications for cement and concrete with lower carbon intensity are commonplace. Wood-fuel-ready plants such as Texas Lehigh are poised to jump in at the head of the pack to win those bids.

Terry Moore, principal and co-founder
Carbon Shrinks LLC, Austin, Tex.

Postcripts, corrections

July, Chementator, Fast pyrolysis, pp. 11–12: In the description of the fast pyrolysis plant that Ensyn Technologies is building with Tolko Industries in High Level, Alberta, the annual production was cited as 850,000 L. In fact the annual production will be 85 million L. ■

Bookshelf



Ludwig's Applied Process Design for Chemical and Petrochemical Plants. 4th ed. Vol. 2. By A. Kayode Coker. Elsevier Inc. The Boulevard, Langford Lane, Kidlington, Oxford OX5 1GB, U.K. Web: elsevier.com. 2010. 961 pages. $223.00.

Reviewed by Kamal Shah, Aker Solutions, Houston

The comprehensive and practical fourth edition of "Ludwig's Vol. 2" is written as a process design manual for the distillation unit operation. Intended for process engineers involved in designing distillation columns, trays and packed towers, the book provides in-depth discussion of design methods not only for distillation, absorption and stripping units, but also for enhanced process units involving extractive, azeotropic and reactive distillation.

Among the interesting and unique features of the book is a detailed discussion of the design impact of tray geometry and details related to the mechanical design of trays and their hardware — information practicing chemical engineers involved with column trays should find useful.

Throughout the book, the author makes good use of equations, graphs, charts, calculations and examples. A broad reference list, appendices and supplements are also included.

This book's (Volume 2) five chapters begin at Chapter 10, which, after discussing foundation concepts, provides guidance on selecting thermodynamic models for simulation using commercially available software, as well as selecting equations of state and K-value methods. Other notable features of Chapter 10 include information on determining thermodynamic properties, methods for carrying out multicomponent equilibrium flash, bubble point and dewpoint calculations, and methods for component separation.

Chapter 10 also includes a rigorous explanation of plate-to-plate calculations using total or partial condensers and multiple-feed and side-stream configurations. In addition, the chapter describes methods for tray sizing and geometry selection, as well as tools for analyzing column performance. Concepts such as heat integration of columns, capital cost considerations and tradeoffs are explained in an easily understandable manner.

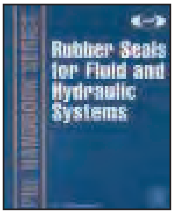
Chapter 11 covers complex petroleum-mixture characterization and discusses the use of fractionation-distillation units to produce different products. This chapter also discusses applications in natural-gas processing.

Enhanced distillation techniques, such as maximum- and minimum-boiling azeotropes, heterogeneous azeotropic distillation, close-boiling-point component distillation, pressure-swing distillation, extractive distillation and reactive (catalytic) distillation, are covered in Chapter 12.

Chapter 13 deals with the impact of mechanical design on tray performance. The chapter covers several tray types and the application, mechanical design and hardware details of each. The chapter devotes space to design methods and specifications to be used during the mechanical design of column tray components, including associated hardware details for proper tray hydraulics and liquid distribution of the feed, side draws and reflux.

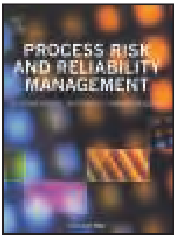
Chapter 14 provides details for various packing types, packing supports, liquid distributors, intermediate packing supports and redistributors. It offers an extensive review of packed columns, as well as generalized pressure-drop charts and equations for sizing columns with random and structured packing.

The book serves as an excellent resource for distillation unit operations that are crucial to the gas-processing, chemical and petrochemical industries.



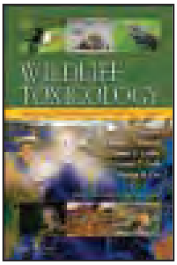
Rubber Seals for Fluid and Hydraulic Systems. By Chellappa Chandresekaran. Elsevier Inc., 30 Corporate Drive, Suite 400, Burlington, MA 01803. Web: elsevier.com. 2010. 160 pages. $149.00.

Process Risk and Reliability Management: Operational Integrity Management. By Ian Sutton. Elsevier Inc., 30 Corporate Drive, Suite 400, Burlington, MA 01803. Web: elsevier.com. 2010. 850 pages. $195.00.



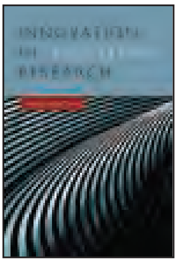
Wildlife Toxicology: Emerging Contaminant and Biodiversity Issues. Edited by Ronald J. Kendall and others. CRC Press, 6000 Broken Sound Parkway, NW Suite 300, Boca Raton, FL 33487. 2010. 340 pages. $119.95.

Thermo-oxidative Degradation of Polymers. By T. Roy Crompton. iSmithers Rapra Publishing Ltd., Shawbury, Shrewsbury, Shropshire SY4 4NR, U.K. Web: ismithers.net. 2010. 136 pages. $200.00.



Advanced Process Control: Beyond Single Loop Control. By Cecil Smith. Wiley, 111 River St., Hoboken, NJ 07030. Web: wiley.com. 2010. 450 pages. $99.95.

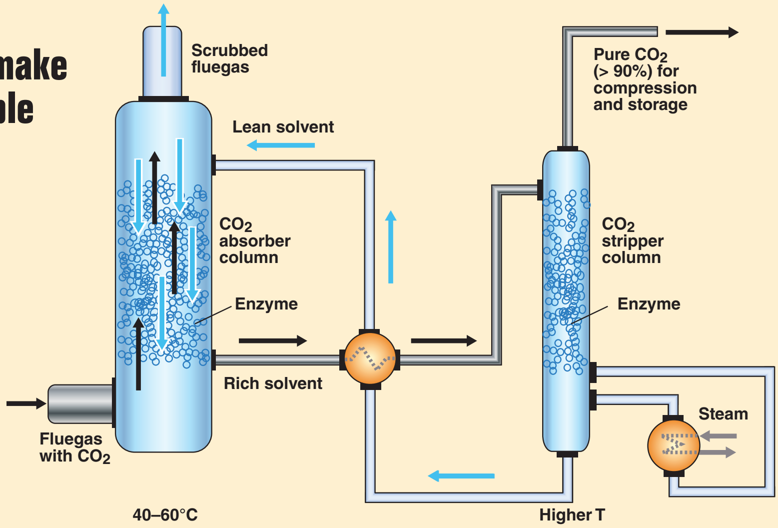
Innovation in Industrial Research. By Paulo de Souza. CSIRO Publishing, P.O. Box 1139, Collingwood, Victoria 3066, Australia. Web: csiro.au. 2010. 152 pages. $44.95.



Advances in Catalysis. Vol. 53. Edited by Bruce Gates, Helmut Knoezinger and Friederike Jentoft. Elsevier Inc., 30 Corporate Drive, Suite 400, Burlington, MA 01803. Web: elsevier.com. 2010. 644 pages. $224.00. ■

Scott Jenkins

Enzyme-based method could make CO_2 capture economically viable



Codexis (Redwood City, Calif.; www.codexis.com) and CO_2 Solution (Quebec City, Canada; www.co2solution.com) have jointly developed a cost-effective method for capturing carbon dioxide from coal-fired power plants.

Solvent-based systems for capturing CO_2 are relatively well-understood, but they have not been widely deployed because they are energy-intensive and inefficient. Solvent-based approaches involve a trade-off between the kinetics of CO_2 absorption by the solvent and the energy required to regenerate the greenhouse gas for sequestration or further use, explains Jim Lalonde, Codexis vice president for biochemistry and engineering R&D.

"You want something that binds CO_2, but not too tightly," he says. For example, the solvent monoethanolamine (MEA) offers efficient CO_2 capture, but requires high energy to regenerate the gas.

The new process depends on two technologies: new forms of the enzyme carbonic anhydrase (CA) that are tolerant of elevated temperatures; and carbon-capture solvents that require less energy to regenerate CO_2.

Codexis is using a proprietary genetic-screening and directed-evolution approach in developing an early prototype of CA that is stable for 30 min at temperatures of 85°C (natural, human CA is unstable above 55°C). The enzyme catalyzes CO_2 absorption into solvents whose properties allow CO_2 regeneration at lower energies. Without the enzyme, these solvents, such as monomethyl-diethanolamine (MDEA), would have unfavorably slow reaction kinetics for CO_2 capture.

In modest quantities, the optimized enzyme accelerates the rate of CO_2 capture in MDEA by about 50 times compared to the rate without enzyme. Once the CO_2 is absorbed, the energy required to drive off the CO_2 is 30% less than the conventional solvent, because of MDEA's sterically hindered molecular structure. The Codexis/CO_2 Solution process is designed to reuse solvent, and is compatible with multiple carbon sequestration approaches.

Codexis is applying its directed-evolution technology to further increase the stability of CA by orders of magnitude and, if successful, will scale up the process to eventually capture tons per day of CO_2 by the end of the current project, Lalonde says. The project is partially supported by a $4.7 million grant from the U.S. Dept. of Energy's (DOE; Washington, D.C.; www.energy.gov) Advanced Research Projects Agency-Energy (ARPA-E).

An attractive way to remove arsenic from water

A magnetic composite based on reduced graphene oxide (RGO) has been developed by a Korean team with an exceptional capacity to remove arsenic from drinking water. The team, headed by professor Kwang S. Kim from the Center for Superfunctional Materials, Dept. of Chemistry, Pohang University of Science and Technology (www.postech.ac.kr), South Korea, claims the composite can achieve nearly complete removal (more than 99.9%) of arsenic — within about 1 ppb — from water. The magnetite-RGO (M-RGO) composite is superparamagnetic at room temperature. It soaks up arsenic when dispersed in water and is then easily removed from the water with a permanent magnet.

Several methods have been developed to remove arsenic from drinking water. For example, arsenic can be removed from drinking water through coprecipitation of iron minerals such as magnetite (Fe_3O_4) nanocrystals. However, those adsorbents are difficult to use in continuous flow systems, such as rivers, due to small particle size and instability — magnetite is highly susceptible to oxidation when exposed to the atmosphere. Therefore, the team decided to synthesize M-RGO due to its large surface area and the stability of the RGO.

The team synthesized graphene oxide via Hummer's method. The graphene oxide was exfoliated in water to produce a suspension of graphene oxide sheets. An aqueous mixture of $FeCl_3$ and $FeCl_2$ was added slowly to the solution, and ammonia solution was added quickly to precipitate Fe^{+2} and Fe^{+3} ions for synthesis of magnetite nanopar-

(*Continues on p. 12*)

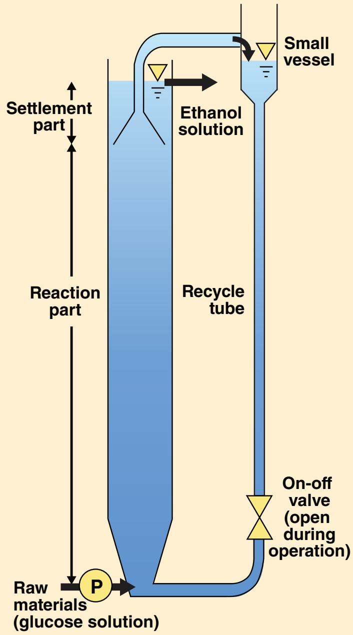
World's largest PDH unit

Lummus Technology (Bloomfield, N.J.), a CB&I company (The Woodlands, Tex.; www.cbi.com), has been awarded a contract by Tianjin Bohua Petrochemical Co. for the license and engineering design of a grassroots propane dehydrogenation (PDH) unit to be built in Tianjin, China. It will be the first PDH plant in China, and upon startup in 2012, will be the largest PDH plant in the world, says CB&I.

The unit will use the Catofin dehydrogenation process to produce 600,000 metric tons per year of propylene. (for more on Catofin and PDH, see Pushing Propylene Production, *CE*, March 2004, pp. 20–24)

Note: For more information, circle the 3-digit number on p. 62, or use the website designation.

A more efficient, less expensive way to continuously make bioethanol



An enhanced reactor technology that boosts the speed of producing bioethanol by a factor of four while decreasing production costs by 25% compared to a stirred batch fermenter has been developed by IHI Corp. (IHI; Tokyo; www.ihi.co.jp). The technology features a cohesive strain of yeast to increase the number density of microorganisms in the reactor, and utilizes the flow created by the carbon dioxide gas released by the fermentation to enhance the contact (mixing) between the yeasts and glucose feed solution. As a result, the continuous process eliminates the need for mechanical agitation and aeration, thereby reducing operating costs, maintenance and reactor size.

In IHI's process, glucose solution is continuously fed to the bottom of the reactor and flows upward by the gas generated by fermentation. The gas is collected by an umbrella-like structure at the top, and then moves into a small side vessel. There, the gas is separated from the fermentation broth (yeast aggregates and liquids), which is recycled to the bottom of the reactor. Ethanol is recovered from a side stream leaving the settlement part of the reactor.

In a pilot reactor (8.6-L reactor volume, 1-m high and 10-cm dia.) operating at its Yokohama facility, IHI has demonstrated the technology with one month of continuous operation. An ethanol production speed of 25 g/Lh was achieved, and the average size of the yeast aggregate maintained at 0.5–1.5 mm.

A new iodine-based catalyst for asymmetric synthesis

Professor Kazuaki Ishihara and colleagues at Nagoya University (Nagoya, Japan; www.nubio.nagoya-u.ac.jp/nubio4/english/main-e.htm) has discovered an efficient, chiral, salt-based hypervalent iodine catalyst that could replace toxic metal catalysts without generating the waste or explosion risks associated with hypervalent organoiodine complexes. The researchers took the radical step of swapping iodine bonded to carbon for an iodite (IO_2^-) or hypoiodite (IO^-) anion as the oxidant, which can be generated from iodide (I^-) by simple oxidants, such as hydrogen peroxide. The team also coupled the (hypo)iodite with a chiral, quaternary-amine counter ion to solubilize the inorganic iodite in organic solvents and provide a chiral environment to control the geometry of the reaction.

In the laboratory, the researchers performed an enantio-selective, oxidative cycloetherification of ketophenols to 2-acyl-2,3-dihydrobenzofuran derivatives — a key structure in several biologically active compounds — catalyzed by in-situ-generated chiral, quaternary-ammonium-(hypo)iodite salts, with hydrogen peroxide as an environmentally benign oxidant (only water and alcohol are generated as byproducts). After recrystallization, an optical purity of 99 ee % is achieved for this reaction, the highest selectivity with the lowest catalyst loading ever seen in asymmetric, hypervalent-iodine catalysis, says Ishihara. The team confirmed that asymmetric synthesis using inorganic iodide catalyst occurred in several hours for 10 mol% catalyst, which is faster than the 17 hours required for the same concentration of an organic iodine catalyst, suggesting the possibility for real industrial use.

Ishihara expects that a variety of oxidative coupling reactions could be controlled by these salts in place of transition metal catalysts, and is aiming to make an intermolecular version — joining two molecules together rather than reacting two sites on the same molecule.

REMOVE ARSENIC FROM WATER

(*Continued from p. 11*)

ticles. The graphene oxide was reduced to RGO by hydrazine hydrate, which was added slowly for 4 hours at 90°C.

In the composite that the team created, the magnetite nanoparticles are well dispersed in the RGO matrix with an average particle size of 11 nm. The nanoparticles are not simply mixed in or blended with RGO, rather, they are entrapped inside the RGO sheets. The composite is especially effective in arsenic removal — compared with bare magnetite — because the graphene sheets greatly increase the number of adsorption sites. Also, the stability of magnetite is increased by the RGO, so that it can be used in continuous flow systems for longer periods.

Nanofiber applications

Last month, DuPont (Wilmington, Del.; www.dupont.com) introduced the first nanofiber-based polymeric battery separator that boosts the performance and safety of lithium-ion batteries. Tradenamed Energain, the separators are said to increase power by 15–20%, increase battery life by up to 20% and improve battery safety by providing stability at high temperatures. The battery separators are made into a web using a proprietary spinning process that creates continuous filaments with diameters between 200 and 1,000 nm.

The company has begun construction on a facility in Chesterfield, Va., to manufacture product for development and commercial sale. Slated to startup in the first quarter of 2011, the facility will initially have the ability to supply 20% of today's hybrid and electric vehicle needs. While the initial uses for the separator are in hybrid vehicle batteries, the nanofiber technology will target other renewable-energy applications as well as a broad range of liquid filtration applications for the biopharmaceutical, microelectronics, and food-and-beverage industries, offering "superior" retention, filter life and flow resistance, says DuPont.

Heat plus light may boost solar energy efficiency

Photovoltaic cells typically operate at 20% efficiency in converting solar energy to electricity. A new system that could boost the efficiency to as much as 60% by combining the light and heat of solar radiation is being developed at Stanford University (Palo Alto, Calif.; www.stanford.edu).

Standard silicon solar cells have two limitations, says Nick Melosh, an assistant professor of materials science and engineering: they absorb only a portion of the light spectrum, rejecting the rest as heat, and their efficiency decreases as the temperature increases. In contrast, the new system will work best with solar concentrators, such as parabolic dishes, at temperatures up to 800°C. "We have come up with a different physical process from standard photovoltaics," he says.

Stanford's system is a solar cell that has two electrodes, separated by a vacuum. The key element is the cathode, which consists of a monolayer of cesium coated on a semiconductor. Both photons and heat promote the emission of electrons from the cathode, a process the researchers call photon enhanced thermionic emission (PETE).

So far, the researchers have tested the concept in the laboratory at up to 400°C, using cesium-coated gallium nitride. GaN was chosen for the tests because it is thermally stable with cesium, says Melosh, although it is "a terrible material" in terms of energy conversion, achieving only 0.1% electron emission efficiency. "The important thing is that we have demonstrated the capture of light and heat simultaneously and that the process improves with increasing temperature," he says. Melosh is now working with more-common solar-cell materials, such as gallium arsenide and silicon, where the challenge is that these materials are less thermally stable with cesium.

FutureGen 2.0

Last month the DOE awarded $1 billion in Recovery Act funding to the FutureGen Alliance, Ameren Energy Resources Co., LLC (St.Louis, Mo.; www.ameren.com), Babcock & Wilcox Co. (B&W; Charlotte, N.C.; www.babcock.com) and Air Liquide Processes & Construction, Inc. (www.airliquide.com) to build FutureGen 2.0, the world's first full-scale, oxy-coal-fired power plant that includes permanent CO_2 capture and storage (CCS). Proposed funding for the so-called FutureGen 2.0 project would supplement the construction and operation of a 200-MW, near-zero-emissions generating facility to be located at Ameren's Meredosia Plant near Jacksonville, Ill. The project is designed to produce clean energy from coal by capturing and storing approximately 1.3-million ton/yr of CO_2.

If regulatory approvals are received, the next steps would be the negotiations of a cooperative agreement with DOE, followed by a six-month initial design and cost analysis. Construction is planned for 2012, with startup targeted for 2015.

This membrane system extracts hydrogen from mixed gas streams

A metal membrane system developed by Eltron Research and Development (Boulder, Colo., www.eltronresearch.com) separates hydrogen from mixed-gas feed streams that result from gasification, steam reforming and petrochemical processes.

Based on a proprietary, dense metal alloy with hydrogen permeability that is coated on both sides with catalyst material, the membrane can be used as a carbon capture method for integrated gasification combined cycle (IGCC) installations. The membrane system enables the capture of 90 to 95% of the CO_2 while simultaneously producing essentially 100% pure H_2.

A hydrogen dissociation catalyst coated on the feed side of the membrane generates atomic hydrogen, which diffuses across the dense membrane. A reduction catalyst coated on the low-pressure side of the membrane regenerates the H_2 gas. In IGCC applications, the membrane-based process results in a mixture of CO_2, steam and a small amount of carbon monoxide on the high-pressure side of the membrane, with pure H_2 on the other. The differential pressure across the membrane creates the H_2 partial pressure necessary to drive the separation.

Damon Waters, Eltron's manager of business development, says the company is ready to pilot-test various applications on mixed-gas streams, since it has addressed contaminant tolerance and lifetime issues and completed the transition to a tubular membrane format.

Eltron recently announced a joint development agreement with Eastman Chemical Co. (Kingsport, Tenn., www.eastman.com) to scale up and pilot-test the membrane on coal-derived synthesis gas at Eastman's coal gasification facility in Kingsport, Tenn. The $8-million development project is supported by a cooperative agreement with the DOE and will culminate with a pilot demonstration in 2012.

Levels of large-areas

Last month, BinMaster Level Controls (Lincoln, Nebr.; www.binmaster.com) launched the MVL multiple scanner system that integrates multiple-point measurement data from two 3DLevelScanners to cover a very wide surface area. Designed specifically for very large bins, the MLV system displays a visual representation of the material surface that shows high and low points in the bin, such as cone up, cone down, side-wall buildup or bridging. Results generated by the 3D software provide data, such as volume percent; maximum, minimum and average levels or distances to product; weight; and historical logs of bin measurements.

A promising way to reduce the cost of producing oil from oil sands

Roughly 80% of the heavy oil in Alberta's oil sands is too deep for open-pit mining, the most common production method at present. Consequently, this oil will have to be recovered by in situ methods, such as steam-assisted gravity drainage (SAGD), whereby steam is injected underground to liberate the bitumen, which is pumped out through a producer well. Each barrel of oil produced requires 3–4 bbl of water, and while most of the water is separated from the oil and recycled, its recovery is energy-intensive (for more details on oil sands processing, see *CE*, February 2009, pp. 19–22).

GE Power & Water (Trevose, Penn.; www.ge.com/energy) and FilterBoxx Water & Environmental Corp. (Calgary, Alta; www.filterboxx.com) believe they can reduce the energy cost by 30–40%. The two companies have signed an agreement to develop a new water-cleanup process that combines GE's evaporation and reverse-osmosis (RO) membrane technologies with FilterBoxx's high-temperature ceramic membrane technology.

Twelve of GE's mechanical vapor-compression produced water evaporators are already installed at five locations in Alberta, says William Heins, general manager of thermal products for GE's Water Process Technologies Div. The evaporator does the final cleanup following oil-water separation processes, producing pure water from a stream that contains about 2–5% solids and up to 1,000 ppm oil.

The new flow scheme promises to reduce energy costs by substituting membranes for the conventional de-oiling processes, followed by preconcentration of the water by an RO membrane. First, a ceramic membrane will reduce the oil content to a few parts per million. The de-oiled stream will pass through an RO membrane system, which will recover approximately 75% of the stream as pure water, leaving only about 25% for evaporation. The companies plan to pilot-test the membrane technologies later this year.

Nylon-6 intermediate

Engineers at Xiangtan University (Xiangtan, China; www.xtu.edu.cn) have developed a simplified process for the ammoximation reaction of cyclohexanone to cyclohexanone oxime, an important chemical intermediate in the production of *sigma*-caprolactam. Caprolactam is the monomer for nylon-6. The new process utilizes a zeolite catalyst to speed the reaction, which has achieved selectivity and conversion rates better than 99%. The reaction uses cyclohexane as a solvent, rather than the more expensive *t*-butanol in the conventional process. The new scheme reduces byproducts, uses less energy, and is more environmentally benign than the current approach. The researchers are working on optimizing catalyst separation.

Food from algae

TNO (Delft; www.tno.nl) and Ingrepro Renewables B.V. (Borculo, both the Netherlands; www.ingrepro.nl) have started a joint-research project to extract food ingredients from algae. Proteins, which account for up to 60 wt.% of algae, could serve as a sustainable alternative to meat. Another 30% is oil rich in omega-3 and omega-6 fatty acids, which could provide a sustainable replacement for fish oil, says TNO. Little is known about the remaining carbohydrate fraction of algae. The partners believe the utilization of algae as food ingredients will be more profitable than algae-to-biofuel.

In the two-year, €1.5-million project — largely funded by the Biorefinery Encouragement Scheme of the Dutch Ministry of Agriculture, Nature and Food Quality — Ingrepro will be responsible for algae production and for setting up the biorefinery process, and TNO will develop technologies for extraction and refining the oil, proteins and carbohydrates as well as developing applications for the ingredients.

Aluminum-fullerene composites

Researchers from the Technological Institute for Superhard and Novel Carbon Materials (Troisk, Russia; www.ntcstm.troitsk.ru) and Siemens Corporate Technology (Munich, Germany; www.siemens.com) have demonstrated that adding small amounts (1–1.5 wt.%) of fullerenes (C_{60}) to aluminum results in a material that is roughly three times harder than conventional composites while being "substantially" lighter. The new aluminum composites may find applications in compressors, turbochargers and engines. ❑

Sugar-derived compounds can be model solidifiers for oil spills

Professor George John and graduate student Swapnil Jadhav at the City College of New York (www.ccny.cuny.edu) are lead authors of a paper on a class of compounds, known as phase-selective gelators, that can selectively solidify an oil phase in water. These gelator compounds may serve as the basis for next-generation efforts in oil-spill remediation.

The amphiphilic compounds are derivatives of open-chain sugars such as mannitol and sorbitol. To make them amphiphilic, a single-step, site-specific-enzyme catalysis method was used to append alkyl chains to the sugars. The amphiphiles preferentially self-assemble in the oil phase of a water-oil mixture to form solid nanostructures, thereby resulting in selective gelation of oil. When tested with diesel fuel, one gelator compound, dubbed Man-8, immobilized diesel equivalent to 32 times its own dry weight. Recovery of the diesel can be achieved with vacuum distillation. Several of the compounds belonging to the same class were found to exhibit ideal properties for an oil-spill solidifier: low-cost synthesis; high environmental compatibility; selective and efficient gelation of oil in the presence of water at ambient temperatures; ability to facilitate oil recovery, and re-usability. ■

Siemens

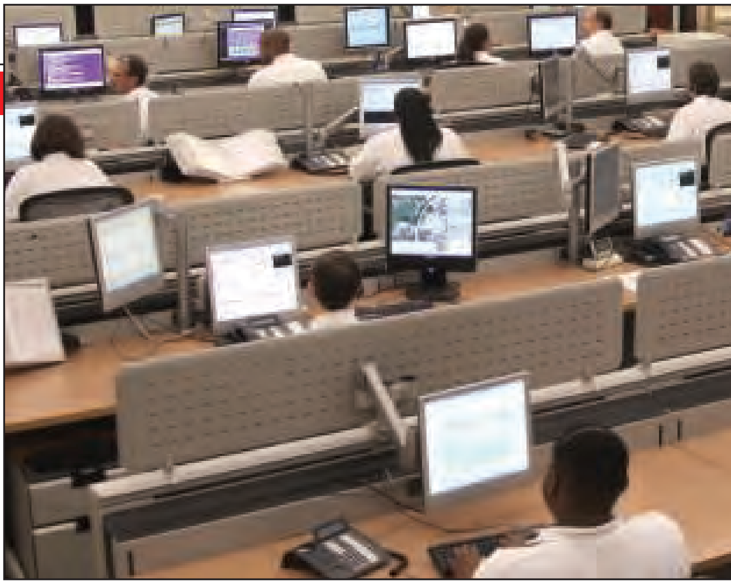


FIGURE 1. Siemens and other industry security experts are seeing a strong trend toward remote, third-party management of security systems. This Siemens center is located in Dallas

SECURING THE CPI

Heightened activity toward CFATS compliance brings the need for integration and a shift of cultural perspective into view

As potential threats continue to make headline news around the world, it is clear that the need for security measures is an ongoing process, and that this process requires vigilance. The chemical process industries (CPI) are considered part of the critical infrastructure in the U.S., and as such are regulated by the U.S. Dept. of Homeland Security (DHS; Washington, D.C.; www.dhs.gov) under the Chemical Facility Anti-Terrorism Standard (CFATS). Already renewed for one year, the standard is again set to expire in October 2010, but all signs are that CFATS is here to stay and the CPI have taken great strides toward compliance.

CFATS implementation

"The chemical industry is well along in implementing the current chemical security regulations, which are rigorous, enforceable and protective of the American public," says Lawrence Sloan, president and CEO of the Society of Chemical Manufacturers & Affiliates (SOCMA; Washington, D.C.; www.socma.com). At the 2010 Chemical Sector Security Summit and Expo held in July, Sue Armstrong, acting deputy assistant secretary for Infrastructure Protection of the DHS, reported on the progress made with CFATS implementation. At the time of the summit, over 38,000 top-screens had been submitted and reviewed; over 6,000 security vulnerability assessments had been submitted and reviewed; over 3,100 site security plans had been submitted and were in review; and DHS inspectors had made over 244 compliance assistance visits. As of June, almost 5,000 facilities were covered by CFATS with rankings in the top four tiers. Over 4,000 of these facilities were given final tiers and the rest were preliminarily tiered facilities (for more on CFATS, "tiers" and other CFATS-related terms see Chemical Plant Security, *Chem. Eng.*, pp. 21–23, September 2009).

Revisions being considered. Even as compliance moves forward, several aspects of CFATS are under scrutiny by the U.S. Congress. One main issue is a proposed revision to mandate use of inherently safer technology (IST). Other areas of discussion relate to broadening the scope of the standard to include, for example, water-treatment plants. Congress is considering multiple bills in the House of Representatives and Senate to either reauthorize the existing standards for a limited time or to significantly alter them.

In late July, legislation that would make the existing CFATS standard permanent took a step forward with bipartisan support in the Senate Homeland Security and Government Affairs Committee. The legislation would extend the existing CFATS by three years, to October 2013. Both SOCMA and the American Chemistry Council (ACC; Arlington, Va.; www.americanchemistry.com) support this extension of the current standard.

Referring to the recent Senate committee's actions, Sloan says "We thank Senator Collins for this extension, which gives chemical manufacturers and DHS valuable time in which to continue to strengthen protections against attack and more fully assess the effectiveness of the entire CFATS program." The next step in the legislative process for this bill is consideration by the full Senate.

Harmonization. While CFATS is the main regulatory agent for CPI security, there are a number of exemption categories under which facilities must comply with other governing bodies. One example is that some facilities fall under the Maritime Transportation Security Act (MTSA) regulations. For some of these facilities, there is concern about how to comply with overlap among various government agencies, and this concern was expressed during a question-and-answer period at the July Chemical Sector Security Summit. This concern is recognized and being addressed by an effort to create multi-agency harmonization through working group initiatives, such as the MTSA/CFATS Harmonization group.

Security experts

Security in the CPI did not start with CFATS. As ACC president and CEO Cal Dooley states, "Since 2001, ACC members have helped lead the way on chemical security, well ahead of government regulation. They have invested more than $8 billion on facility security enhancements under ACC's Responsible Care Security Code."

Yokogawa

FIGURE 2. The integrated field wireless gateway (left), EJX-B Series differential pressure and pressure transmitter (center) and YTA Series temperature transmitter are Yokogawa's field wireless devices based on the ISA100.11a standard

Even so, there has been much activity in CPI security recently, mostly due to CFATS and perhaps partly due to the greater visibility and interest given to security and the corresponding increase in available resources.

Ryan Loughin, director of the chemical & energy solutions of ADT Advanced Integration (Norristown, Pa.; www.adtbusiness.com/petrochem-ce) says that "Just in the last 30–60 days there has been a lot of movement from DHS, so there has also been a lot of movement from the CPI," referring to requests for security system design, awarding of contracts and more. Companies are asking for help in designing their site security plans (SSPs) and then redesigning them when rework is needed as feedback from the DHS is received, explains Loughin.

For CPI facilities seeking help, there are a number of security-expert companies, such as ADT and others, that offer assistance with designing, building and managing security systems. And these companies have developed expertise with CFATS so that they can, for example, specifically address what a facility might do to meet the requirements of the risk-based performance standards (RBPSs) that are established in CFATS. "One of the beautiful things about the RBPSs," says Michael Saad, senior director consulting services with Huffmaster Crisis Response (Troy, Mich.; www.huffmaster.com), "is that they leave it to the facility to determine how to achieve the level of security performance required. But that is a sword that cuts both ways. It also requires the facility to determine how to create layers of integrated security." This is where the security experts can help.

Managed services and other trends. At a briefing given on August 10 at Siemens AG's (Munich, Germany; www.siemens.com) U.S. headquarters for their Security Solutions Business Unit (Buffalo Grove, Ill.; usa.siemens.com/buildingtechnologies), Carey Boethel, vice president and business unit head for Security Solutions, explained two trends that are currently shaping security strategies. The first is a proliferation of physical security information management (PSIM) and command-and-control centers. This refers to command-and-control centers that work toward an efficient information flow during emergency situations to ensure a unified command and intelligent response to threats — a control center at a police department was given as an easy-to-understand example. Formerly, only very large businesses were interested in these centers due to the expense, but more recently that is changing.

The second trend is hosted or managed services. This refers to hiring an outside company — a security expert — to monitor the incoming security data (from video surveillance, for example, but also a wide-range of other information) and initiate actions when needed. As Boethel explains, "Hosted applications once focused on small and medium-sized businesses, but are now entering an enterprise-class phase where large multinational companies are embracing the technology." In fact, Boethal expects that managed video services industry-wide will have more than a 15% compounded annual growth rate. An interesting conclusion can be drawn from these two trends — the distinction between "high-end" and "low-end" security system users is going away. Large, mid-sized and small facilities are exploring all options for their site security plans.

Phil Atteberry, director of managed security services at Siemens, manages both of Siemens' remote-monitoring centers in Dallas, Tex. (Figure 1) and Beltsville, Md. He has seen a "huge trend" toward managed services with about an 8% increase from 2007–2010 in third-party security monitoring. Atteberry sheds some light on the reasons for this increase when he says "Before it was a single-alarm system, but now it is more enterprise-based … one focal point can monitor the total situation awareness of the facility." Advantages of third-party security monitoring include having an expert, who best understands the incoming security data, monitor it; reducing or eliminating the need for on-site monitoring personnel and the associated costs; and reducing the potential that an event might be missed due to a lapse of attention to a video screen, for example, during a long period of inactivity on the screen. Since remote centers monitor a number of sites, there is inherently more activity being monitored and therefore more stimulation to keep personnel attentive.

The increase in requests for remote monitoring is a trend that has been noted by a number of security experts. ADT's Loughin sees more requests for third-party video and alarm monitoring for their special operations monitoring facility in Aurora, Colo., and cites cost-effectiveness as a strong reason for this movement. Saad also notes that an increase in demand for Huffmaster's interactive remote-video-monitoring services led Huffmaster to renovate and expand the space and resources devoted to those services.

Securing process control

For the CPI, securing process control systems is a vital part of any security plan. And, the CPI has been very actively involved in leading-edge efforts in cyber security. An example of this is the Roadmap to Secure Control Systems in the Chemical Sector (www.us-cert.gov/control_systems/pdf/ChemSec_Roadmap_First_Edition.pdf), which is a document published in September 2009 that presents a security vision for industrial control systems in the CPI, proposes a comprehensive plan for implementation, and identifies milestones over a ten-year period. Preparation of the roadmap was truly a cooperative effort that included the DHS and a substantial number of volunteers from CPI companies.

Citing the roadmap and other efforts in cyber security standards, Ernest Rakaczky, portfolio manager of control system cyber security for Invensys Operations Management (Plano, Tex.; www.iom.invensys.com) says "In a lot of ways, the chemical sector is leading the way." Rakaczky stresses the importance of control and IT experts working together toward the common goal of security. He says that typically, firewall technology is changing faster than controller technology. With a core competence in control, Invensys builds its control systems so that they can easily be updated with the latest security systems. This built-in ability to update the control system allows the user to take advantage of the newest technologies without large investments.

Threats to control systems can come from a variety of sources. Firewalls are very effective at preventing intrusion. People with access to the control system, however, are a credible threat, whether through accidental means (such as unwarily using personal devices or USB sticks), or through intentional means (such as by a disgruntled employee for example). "A malicious insider is the tough threat," says Graham Speake, principal systems architect for Yokogawa Corp. of America's (Newnan, Ga.; www.yokogawa.com/us) IA Global Strategic Technology Marketing Center in Dallas, Tex. Threats to control systems are "no longer theoretical" says Speake, citing the recently discovered Stuxnet worm that was distinctly targeted to attack a specific control system. "One way to increase security," says Speake, "is to build more security into products so that no matter how you put them together they are still secure." Yokogawa says it has taken a step in that direction by developing the world's first field wireless devices based on the ISA100.11a industrial wireless communications standard (Figure 2).

What makes the Stuxnet worm such big news in the control community is not just that it was deliberately made to attack control systems, but that it is highly sophisticated — enough so that it attacks a previously unkown vulnerability and it wraps libraries to hide PLC programming changes. "The worm is a wake-up call. A lot of operations people and even security decision makers had still been on the page that malware is only an incidental threat to control systems and physical processes, and that sophisticated threats … were not something they had to worry about. All that has changed with Stuxnet," says Andrew Ginter, chief security office with Industrial Defender, Inc. (Foxboro, Mass.; www.industrialdefender.com).

A basic principal of cyber security plans offered by Industrial Defender is Defense-in-Depth (Figure 3), which presumes that no single mode of security mitigation is impenetrable and therefore several layers are needed. The company's approach includes three major categories including: intrusion prevention, which is applied at network perimeters with a firewall, and within servers and end-point devices via the recently introduced Host Intrusion Prevention System (HIPS); access management, which provides rigorous authentication and authorization before allowing access through the perimeter; and monitoring and response, which integrates intrusion detection, performance monitoring and event management.

Third-party management. An interesting shift is occurring in that more companies are looking for outside security management of not only physical security systems, but also cyber security systems. Ginter explains that while there has been a reluctance to let information out to third parties, companies are getting more comfortable with this through cloud computing and "software as a service", where computers and functionality are rented. He says that some CPI customers have said that "If you've got a good security posture now and you want to take a step up, outsource it." Their reasoning is that industrial-control-security experts, such as Industrial Defender, understand the system and follow the rules (set up by the user) since that's what they get paid to do. Industrial Defender has a remote control center at its headquarters in Foxboro, Mass.

Invensys' Rakaczky says, "One element where there is a big future is managing security. The overall success of security is not [a question of] how many firewalls or security devices I put in, but once I do this, how do I manage it?" He sees a goal to be learning how to address an attack and how to manage it so that a user can work through the attack (and not shut down). And Speake, who fairly recently joined Yokogawa coming from a CPI company, says that with his CPI background, when it comes to managing the security systems he would choose to outsource it. He cautions, however, that you would have to be careful about who does it and where.

FIGURE 3. Industrial Defender's Defense-in-Depth solution takes a multi-pronged approach to security

A cultural shift

With CFATS implementation well underway, and cooperative working groups, such as those involved with the Roadmap to Secure Control Systems in the Chemical Sector, and those working on standards to improve wireless security, there is a heightened level of awareness about security — both physical and cyber — in the CPI. Most agree that the typical CPI facility has a well-established safety culture where, as Rakaczky puts it "everybody in the plant has a stake in ensuring that everyone is safe." He says that this type of culture that drives safety is getting started on the security front, and that "training, policies and procedures, and managing everyday activities will have the biggest influence on cyber security."

Andrew Wray, senior global marketing manager with Honeywell Process Solutions (Phoenix, Ariz.; www.honeywell.com), another leader in expert security systems, has noticed a "cultural shift in a plant environment where everyone feels more a part of security." Whereas people have previously viewed certain surveillance items, such as video cameras, an intrusion on their privacy, Wray says that now "People believe they have a right to feel secure and safe, and they see video surveillance as a benefit to that end." ■

Dorothy Lozowski

FROM SEA TO SHINING SEA

Veolia Water Solutions and Technologies

FIGURE 1. Pictured is a Chevron facility in San Ardo, Calif., where Veolia's OPUS system has been operating

Desalination technologies are improving to make seawater and brackish water treatment more efficient and cost effective for industry

Although the municipal water demand is considerably greater than that of industry, many of the municipal needs are addressed by relatively basic technologies, while industrial requirements can only be met with advanced treatment options. And, as industrial water demands continue to grow due to regulatory restrictions and mandates, water treatment providers are developing innovative desalination technologies, which allow saltwater and brackish water supplies to be cleaned up in a more cost effective, energy efficient manner.

"Municipalities are beginning to reserve fresh water supplies for the population, which means industry is now required to find alternative water sources, such as seawater and brackish water to support manufacturing processes," notes Gary Martin, segment general manager of water and wastewater solutions, with Siemens Water Technologies (Warrendale, Pa.). "We're finding this especially true in applications for the power, chemical and mining industries where we are increasingly being asked to find solutions for retrofits, expansions, upgrades or new installations."

Siemens Water Technologies

FIGURE 2. This photo shows an installation of an RO high-pressure pump and the energy recovery devices (in the background)

Typically brackish water is used more broadly in industry, but that doesn't preclude the use of desalination of seawater as a key technology solution to the overall issue of industrial water scarcity around the globe. "Just as you have multiple ways to generate electricity, such as coal, wind, sun and gas based on geography and atmospheric conditions, the same can be said of water," notes Joseph Such, general manager of reverse osmosis and electrodialysis systems with GE Power & Water (Trevose, Pa.). "We can clean up water using different technologies such as desalination of seawater or reuse of brackish water. Clearly, with water scarcity, we are looking at all the different levers we can pull to assist with this issue, and that may include desalination of saltwater if the customer is near the sea, or reusing municipal or industrial wastewater if the customer is inland."

He continues to say that this will remain a persistent issue for industry even in areas where fresh water is plentiful due to mandates that prohibit facilities from disposing of wastewater, permits being withheld due to high water use or by economic drivers such as the increasing cost of fresh water. "In any of those situations, desalination will undoubtedly be part of the solution."

Industrial technologies

Although municipal- and industrial-desalination needs share some common technologies in that most advanced treatment technology uses membranes for removal of suspended solids and dissolved solids, in municipal applications these two methods are usually sufficient to meet the quality requirements. In industrial applications, however, the water often needs additional treatments, such as

FIGURE 3. GE Water's SeaPRO seawater RO desalination system offers flowrates of 100 to 2,500 m^3/d (20 to 460 gal/min)

ion exchange or electrodeionization, or specialized membranes to increase the quality beyond potable standards.

For example, if desalinated water is being used to feed a high-pressure boiler, more salts will need to be removed because under high pressures and high temperatures, the salts tend to corrode the materials inside the boilers. So in addition to the reverse osmosis (RO) systems, which will remove about 97% of the salts, an additional process, or polishing step, such as ion exchange media or electrodeionization, may be added on the back end.

Another factor for industrial desalination may be the temperature of the water. Many processors try to reuse condensate. This fairly high-temperature water is cleaned up and put back at the front end of the boiler. If industrial desalination or brackish-water reverse-osmosis technologies are employed, it is likely that the best, most energy efficient solution will be to keep as much of the temperature in the water as is possible and feed it to the boiler at the higher temperature so it doesn't have to be reheated. This would require advanced, high temperature membranes.

Water treatment providers are also working on membranes that are more efficient. "Everyone is looking to get more water for less energy and less cost," notes Ian Lomax, global market manager for desalination, with Dow Water and Process Solutions (Minneapolis, Minn.). "Flux is the amount of water you get for a given area for a given pressure, and over the last 20 years, the flux of the membrane has improved by about two to three times. And we're still looking for the next level. Today we're working on products that we expect will achieve higher flow for lower energy than the products we have today."

GE Water and Process Technology

FIGURE 4. The GE PROPAK is an integrated ultrafiltration (UF) and reverse osmosis (RO) system for providing high quality water from high variability surface water

Energy efficiency

However, advanced membranes aren't the only way to skin the energy efficiency cat. Energy recovery devices, which capture some of the waste energy from the process and recycle it, are also helping to increase the energy efficiency of desalination processes.

"These devices are especially important in seawater desalination because there is so much salt in the water that it must be driven at high pressures at the input of the membrane system," explains GE's Such. Typically the input pressure is about 900 psi and the output pressure is roughly 800 psi. "Energy recovery devices can be put there, almost like a turbo charger on a car, to capture the energy that would otherwise be put down the drain and allow it to prefeed the high-pressure pump on the front end. Now instead of taking it from feed pressure to 900 psi, it only needs to go from 800 to 900 psi."

When energy recovery devices are combined with membranes that require less energy, the driving pressure up front becomes less and less, which lowers both the cost of the pump and the amount of energy required to operate the pump.

"It is a multi-pronged approach when you look at energy consumption in a membrane plant," says Such. "It isn't any one component, but rather all the components used that create a robust, highly effective and more efficient desalination system."

For this reason, many water treatment providers are offering pre-engineered modular system, says Siemens' Martin. "When matching standard re-

SOLAR DESALINATION

Worldwide, more and more people are obtaining drinking water either from the sea or from increasingly salty inland sources. Analysts at Global Water Intelligence, an industry service, estimated that in 2008, desalination facilities around the world produced nearly 12-billion gallons of water each day. This amount is expected to more than double by 2016. The bulk of this amount is attributable to the Arab countries around the Persian Gulf. Other countries such as Spain, France, Greece and Italy are also turning to desalination. While most large desalination plants are fueled by oil or gas, smaller and medium-sized plants can run partly or even entirely on renewable energies.

"Often people in these plants don't know how reliable and efficient solar-powered desalination plants, or wind-powered and, perhaps someday tidal-energy-driven plants, work," says Marcel Wieghaus of the Fraunhofer Institute for Solar Energy Systems (ISE; Freiburg, Germany; www.ise.fhg.de). The European ProDes initiative, of which ISE is a member, intends to change that by conducting a study of examining how renewable energies can be harnessed more effectively in the future to transform seawater and brackish water into drinking water.

ProDes, which stands for Promotion of Renewable Energy for Water Production through Desalination, has established an array of processes for desalinating seawater and brackish water. Currently most European desalination plants rely on reverse osmosis, where high-pressure and semi-permeable membranes separate water from salt and unwanted organic constituents. Plants operating on conventional energy forms deliver around 400,000 m^3/d of drinking water. When it comes to desalination plants running on renewable energy, the spectrum ranges from simple solar distillation plants with a capacity of a few liters a day to wind-powered reverse osmosis plants capable of desalinating nearly 2,000 m^3.

Ultimately the best technology for the task depends on the salinity of untreated water, the local infrastructure and the quantity of water required. "The more remote the location, the more worthwhile and profitable it is to use plant systems run on renewable energy and to set up a water treatment operation that is not dependent on an external energy supply," explains Weighaus. He and a team of researchers have installed solar-powered desalination plants on the Canary Islands of Gran Canaria and Tenerife.

In a similar vein, IBM (Armonk, N.Y.) and the King Abdulaziz City for Science and Technology (KACST) (Riyadh, Saudi Arabia) are collaborating to create a water desalination plant powered by solar electricity, which could significantly reduce water and energy costs.

A new, energy efficient desalination plant with an expected production capacity of 30,000 m^3/d will be built in the city of Al Khafji to serve 100,000 people. KACST plans to power the plant with the ultra-high concentrator photovoltaic (UHCPV) technology that is being jointly developed by IBM and KACST. This technology is capable of operating a CPV system at a concentration greater than 1,500 suns. Inside the plant, the desalination process will hinge on an IBM-KACST developed technology, a nanomembrane that filters out salts as well as potentially harmful toxins in water while using less energy than other forms of water purification.

According to KACST scientists, the two most commonly used methods for seawater desalination are thermal technology and reverse osmosis, both at a cost ranging from 2.5 to 5.5 Saudi Riyals per cubic meter. Combining solar power with the new nanomembrane, the project may significantly reduce the cost of desalinating seawater at these plants.

"Currently Saudi Arabia is the largest producer of desalinated water in the world and we continue to invest in new ways of making access to fresh water more affordable," says Dr. Turki Al Saud, vice president for research institutes, KACST.

Because over 97% of the world's water is in the oceans, turning salt water into fresh water cost effectively and energy efficiently offers tremendous potential for addressing the growing worldwide demand for clean water. One of the most efficient means of desalination is reverse osmosis. But there are obstacles to unlocking this reserve — principally biofouling, degradation by chlorine and low flux challenges. The joint research focuses on improving polymeric membranes through nanoscale modification of polymer properties to make desalination much more efficient and much less costly.

"Our collaborative research with KACST has led to innovative technologies in the areas of solar power and of water desalination," says Sharon Nunes, vice president, IBM Big Green Innovations. "Using these new technologies, we will create energy-efficient systems we believe can be implemented across Saudi Arabia and around the world." ❑

verse osmosis modules with standard pre-treatment and post-treatment products, it has the multiple effect of reducing energy use, reducing costs, increasing availability through the use of products that are proven to work together and shorter schedules and start up times."

Brad Biagini, a product manager with Veolia Water Solutions and Technologies (Cary, N.C.) agrees that all-in-one treatment technologies are efficient solutions. For example, the Amdro from Veolia, which he says was initially developed for water treatment in the phosphate fertilizer industry, is a reverse osmosis technology operated in double-pass mode. By combining the company's Actiflo high-rate clarification process with filtration, RO and ion exchange, the technology generates high-quality effluent with minimum pre-treatment requirements. Similarly, the company's OPUS technology uses a reverse osmosis process operated at an elevated pH. By combining a proprietary high-rate chemical softening process, known as Multiflow with filtration, ion exchange and reverse osmosis, this technology generates high quality water with a low waste volume.

And GE Water's Propak NA Series, which was designed for industrial applications in boiler feed, combines ultrafiltration hollow-fiber membranes to take out solids with spiral wound RO membranes to take out dissolved salts on a single skid. "Being on one skid saves on footprint, and because they aren't buying two separate systems, it also saves on capital costs. Operational cost savings can also be had because the systems are optimized to work together." ■

Joy LePree

WATER TREATMENT PROVIDERS

BWA Water Additives www.wateradditives.com
Dow Water and Process Solutions www.dowwaterandprocess.com
GE Water and Process Technologies www.gewater.com
IBM www.ibm.com/smarterplanet/water
Siemens Water Technologies www.water.siemens.com
Veolia www.veoliawaterst.com

ChemInnovations 2010 Conference & Expo

The first ChemInnovations 2010 Conference and Expo (www.cpievent.com) is designed to provide engineers in the chemical process industries (CPI) with tools and knowledge for solving today's complicated processing challenges. Scheduled to take place at the Reliant Center in Houston October 19–21, ChemInnovations is expected to attract over 150 exhibitors and more than 2,000 attendees.

ChemInnovations is focused on fostering innovative thinking and on delivering the innovative technologies and approaches that are vital in addressing current processing challenges. The event was put together to help attendees anticipate market and regulatory trends in the CPI.

Conference keynote speakers will address various aspects of innovation, including how to cultivate innovative thinking and support innovation while meeting the regulatory demands placed on the CPI.

The ChemInnovations conferences are organized into seven tracks, with track titles including the following:

- Business insight for engineers
- Process design and operations — nurturing your plant production
- Energy efficiencies and the use of alternative energy sources
- New horizons in automation
- Environmental, health and safety — let's talk regulations and operations

The ChemInnovations 2010 event program also features poster sessions, CPI training seminars, workshops and a host of special activities.

CHEMENTATOR LIGHTNING ROUND

Further in keeping with the innovation theme, ChemInnovations 2010 will showcase a unique track called the Chementator Lightning Round. This track parallels a monthly editorial department in *Chemical Engineering*. Using a fast-paced, interview-style format, the Chementator Lightning Round will provide a look at newly proven technologies and approaches that could lead to breakthroughs in CPI applications. The interviews will be led by the *Chemical Engineering* editorial staff. The following technologies are among those to be presented within this track at ChemInnovations 2010. The date and time of each presentation follow each writeup.

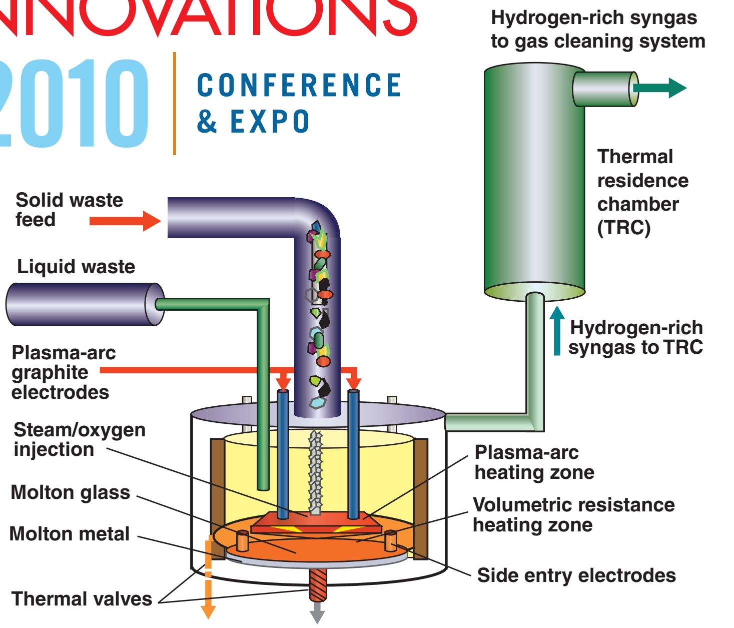


FIGURE 1. InEnTec's PEM uses a direct-current plasma arc to generate syngas from waste and other materials

Source: InEnTec Chemical

Plasma-enhanced gasification offers improved waste disposal

Plasma-enhanced melter (PEM) gasification technology developed by InEnTec Chemical LLC (Fleming Island, Fla.; www.inentecchemical.com) employs a direct-current plasma arc and an alternating-current glass melter to generate synthesis gas (syngas) from hazardous and other wastes (Figure 1).

As waste is fed into the primary vessel, it contacts the plasma and is immediately broken down into its atomic and molecular constituents. The dissociated products of organic waste recombine to form the components of synthesis gas, while inorganic materials are entrained or dissolved in the glass phase. Since steam and oxygen are also fed into the PEM gasification reactor, the PEM essentially conducts an almost instantaneous steam-methane reforming reaction.

The company says the syngas produced is of high purity, and the PEM works well on nearly all forms of biomass and waste material, including hazardous waste from chemical plants, municipal waste, non-hazardous industrial waste and medical waste. The initial commercial site for this technology is operating at Dow Corning's Midland, Mich. site. In addition to being convertible to other products, the syngas produced by the PEM can also be used as a fuel to produce energy, a scheme with attractive energy balances, the company says. — *Oct. 21, 12:30 PM*

A steam thermocompressor with improved efficiency

Through a careful redesign informed by computational-fluid-dynamics (CFD) modeling, a research team at Kadant Johnson Inc. (Westford, Mass.; www.kadant.com) has developed a steam jet thermocompressor that improves performance and energy efficiency compared to conventional units.

Thermocompressors use high-pressure steam to entrain flow from a lower-pressure steam source in order to boost pressure for reuse. By optimizing the thermocompressor geometry, entrainment-ratio improvements of 15 to 25% over conventional designs have been observed. Thermocompressors have been applied widely in the CPI to boost low-pressure steam, but are often oversized and operate at higher differential pressures than are needed. The results are energy waste, operational problems and poor control. — *Oct. 20, 11:50 AM*

A new refractometer compensates for drift

A novel refractomer developed by Flexim GmbH (Berlin, Germany;

Note: For more information, circle the 3-digit number on p. 62, or use the website designation.

www.flexim.de) compensates for drift and deposit sensitivity in inline process measurements of refractive index, overcoming a key disadvantage of conventional instruments.

Rather than measuring the critical angle of refraction, the new refractometer sets up a differential measurement based on a method that measures light transmitted through a bi-prism.

A parallel light beam crosses the fluid to be measured, and is refracted and split at the surface of the measuring prism. The two intensity peaks from the split beam are detected on a charge-coupled device (CCD) sensor that is positioned in the focus of an optical lens. The distance between them is correlated to the refractive index of the fluid through Snell's Law (ratio of the sines of the angles of incidence and refraction equals the ratio of the velocities in the two media).

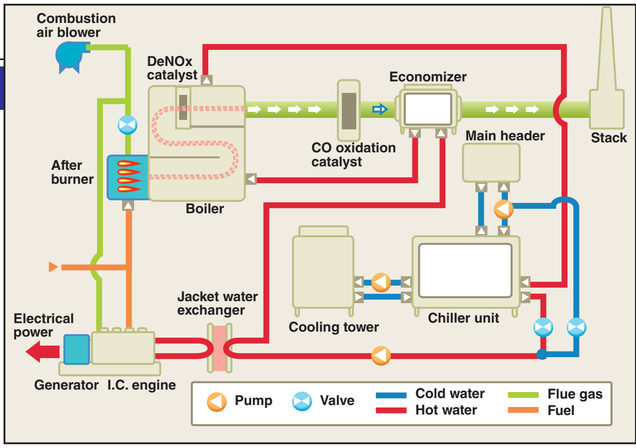
While the critical angle approach can be useful in the laboratory, its value is diminished somewhat in process applications because only the material at the prism surface contributes to the measurement. This characteristic can be problematic because measurements are sensitive to particles adhering to the prism surface, and are not applicable for low-flow conditions or highly viscous materials. — *Oct. 20, 3:30 PM*

Cogeneration process has high thermal efficiency, low emissions

The Ecoces Cogeneration system produces electrical and thermal energy with thermal efficiencies of 90 to 92% and achieves exhaust emissions of less than 5 ppmv for NOx and 10 ppmv carbon monoxide. The cogeneration system can be applied to both lean-burn and rich-burn internal-combustion (IC) engines.

To achieve higher thermal efficiencies than conventional systems, the Ecoces system combines engine exhaust from an IC-engine-driven generator with combustion air (Figure 2). The controls allow the auxiliary burner to fire a heat-recovery steam generator at near stoichiometric oxygen-to-fuel ratios, maximizing the overall thermal efficiency and minimizing emissions.

"This approach to cogeneration provides for a means to produce steam at varying rates, independent of electrical load," comments technology developer Ronald Bell.



Source: Ronald Bell

FIGURE 2. To achieve high thermal efficiencies, the Ecoces cogeneration system combines engine exhaust from a generator with combustion air

Bell, an industry consultant, originally patented the technology in 1991, then optimized the control system in 2005, and worked with General System Co. Ltd. (Korea) to set up a commercial demonstration in 2006. — *Oct. 20, 11:50 AM*

Catalyst system allows high-efficiency propriolactone production

A process under development by Novomer (Waltham, Mass.; www.novomer.com) utilizes a novel aluminum-based catalyst system that allows carbon monoxide and ethylene oxide to react to form propriolactone with greater than 99% efficiency.

Propriolactone is a versatile chemical intermediate that can be converted to acrylic acid, acrylate esters, 1,3-propanediol and polypropriolactone (PPL), a polymer that has similar properties to polyethylene and polypropylene, but is biodegradeable. The new catalyst system, discovered by Cornell University (Ithaca, N.Y.; www.cornell.edu) professor Geoff Coates, enables an economically viable route to PPL, which has not yet been made on a commercial scale.

In Novomer's process, the CO, ethylene oxide and process solvent are combined in a reactor with the catalyst under mild reaction conditions. After formation of propriolactone, the catalyst is separated from the product and recycled with the solvent.

Novomer says an ethylene-oxide-based route to acrylic acid has strategic advantages over current technology, which is based on propylene oxidation. Further, the company says in 2009, its process was determined to have a cost advantage of more than 30% over existing acrylic acid processes.

Novomer recently completed construction of a continuous pilot unit to verify the catalyst lifetime in a closed loop system. Novomer is seeking partners to commercialize the technology. — *Oct. 19, noon*

Plate reactor can replace batch processes with continuous ones

Alfa Laval's (Lund, Sweden; www.alfalaval.com) ART plate reactor enables some batch processes in pharmaceutical production, as well as fine and specialty chemicals, to convert to continuous operation. The ART reactor uses channeled plates to integrate the mixing channel with heat transfer surfaces, creating a modular, continuous reaction system.

The ART plate reactor consists of a series of cassettes with specially designed channel plates at their cores. Machined into the plates are process and utility channels, which are designed to optimize mixing and heat transfer performance. Each cassette has process inlet and outlet ports, as well as secondary ports through which reactants may be added or monitoring devices may be inserted. Multiple cassettes are typically stacked and held together in a frame.

"Continuous reactors ... will not replace all batch reactors," Alfa Laval explains, "but in cases where the chemistry is limited by the batch reactor, or where safety is an issue, the ART plate reactor can help a great deal."

Two plate-reactor models are commercially available, covering the range of laboratory trials to production of up to 1,000 ton/yr. Standard plate reactors are constructed with stainless steel or Hastelloy, and can operate at temperatures between –60 and 200°C without any reactor changes. Design pressure ranges from full vacuum to 20 bar. — *Oct. 19, noon*

Circle 21 on p. 62 or go to adlinks.che.com/29254-21

Non-continuous hydrogen production from boron hydride

Boron hydride can be used effectively as a source of on-demand hydrogen for cases such as at plant startup. The technology is well developed for small applications, such as automobile fuel cells. Intratec (Rio de Janeiro, Brazil; www.intratec.us) has developed a way to enlarge the scale for this approach.

Current methods for generating industrial hydrogen from hydrocarbons at large scale are not likely to be cost-effective if a non-continuous source of hydrogen is necessary. Further storage of high-pressure hydrogen at large scale can present safety concerns. Intratec has worked out engineering concepts, process technology, chemistry and economics of producing non-continuous hydrogen from boron hydride.— *Oct. 19, 3:45 PM*

Make fine emulsions with ultrasound

Hielscher Ultrasonics GmbH (Teltow, Germany; www.hielscher.com) has developed a new method for producing finely dispersed emulsions using ultrasound on an industrial scale.

The company's high-power ultrasonic processors (Figure 3) generate highly intense sound waves that propagate through liquids and result in cavitation bubbles. The cavitation applies mechanical stress on the surface boundary of immiscible liquids and creates small droplets that are dispersed in the other liquid. Ultrasound can achieve average droplet sizes of less than 1 μm.

Emulsifying agents that efficiently stabilize droplets are added to maintain the final size distribution at a level equal to that immediately following disruption by the ultrasonic waves.

Hielscher's ultrasonic equipment converts electricity into cavitation within the liquid at 80–90% efficiency. And because cavitational forces place a great deal of stress on the liquids, fine dispersions and small particle sizes are possible at lower energies. — *Oct. 21 noon*

Thermal oxidation process features low NOx at high temperature

A research team at John Zink Co. (Tulsa, Okla.; www.johnzink.com) has developed a new thermal-oxidation process, called Tangent, that is capable of producing oxides of nitrogen (NOx) at concentrations of less than 1 ppm in high-temperature (1,800 to 2,300°F) thermal-oxidation equipment.

Tangent works by using highly turbulent mixing to achieve a uniform blend of gaseous fuel and air. "The resulting combustion reaction provides the heat necessary to maintain the chamber temperature and sustain the process," says Chuck Baukal, director of the John Zink Institute.

Establishing a uniform fuel-air mixture eliminates the large temperature gradiants within the combustion process that are typical of flames and that can result in thermal NOx formation. The operating temperature range of the process ensures that carbon monoxide is not produced, notes Baukal.

In contrast to most current approaches to reduce NOx levels, the Tangent system does not require any heat-transfer-media, packing or preheated air to work. Tangent uses a simple refractory-lined vessel.

The Tangent process has been demonstrated at the John Zink test facility, and is available commercially. — *Oct. 21, 12:30 PM*

Hielscher Ultrasonics

FIGURE 3. Hielscher's ultrasonic processor generate high-intensity sound waves that result in cavitation

An inline moisture control sensor permits automatic control

A novel moisture sensor developed by Drying Technology Inc. (Brentwood, N.Y.; www.moisturecontrols.com) that operates inside dryers, ovens and kilns improves moisture control of products and offers an alternative to near-infrared moisture sensors. Based on a

first-principles mathematical model that relates the moisture of product exiting the dryer to the temperature drop of hot air (after contact with the product and the drying rate), the instrument provides a 30% advantage or more in reducing moisture variation compared to conventional means of moisture control.

The control system adjusts the dryer to stay near a target moisture content (as determined by laboratory samples or process set points) in response to changes in the feedrate or moisture level of the feed. Since the dual-temperature-sensor instrument can survive the harsh environment of the dryer, it can operate without the time delays required of conventional sensors that work outside the dryer. It can be used on belt, flash, spray, fluidized-bed and rotary dryers. The system can reduce energy costs by 7 to 10% and can help avoid underdried product material. — *Oct. 20, 3:30 PM*

A non-invasive density and viscosity measurement device

A new non-invasive, percussion-based device for measuring the density of process liquids and loose solids is mounted on the outside of tanks, storage vessels and pipes.

The apparatus consists of a striker and receiver module, which is mounted on process equipment, and a data processing unit. The striker/receiver generates vibrations by making contact with the exterior of the vessel and collects vibration information on the interactions between the vessel wall and the content material. The data processor applies mathematical algorithms to the resulting vibration patterns and calculates density and viscosity.

The software-driven technology allows non-invasive and non-radiometric measurements of density and viscosity regardless of vessel geometry or material, says Alexander Raykhman, a co-inventor of the technology, which is being commercialized by Ultimo Measurement LLC (Providence, R.I.; www.ultimompd.com). The instrument is expected be available commercially before the end of 2010. — *Oct. 20, 3:30 PM*

A phosphogypsum-free route to P_2O_5

A new process has been designed to offer a more energy-efficient and environmentally benign approach to making phosphorous pentoxide (P_2O_5) from phosporite (phosphate rock).

In a direct reaction between an acid gas, such as SO_3 and calcium phosphate, P_2O_5 is liberated from the phosphate rock and calcium sulfate is formed. The calcium sulfate is further heated to produce lime and sulfur oxides.

"P_2O_5 can be recovered as phosphoric acid at desired concentrations without having to go through the phosphoric acid step," says process inventor Mohammed Elgafi, chief engineer at EM&C Engineering Associates (Cerritos, Calif.).

Current wet processes for producing phosphoric acid require 2.8 tons of sulfuric acid per ton of P_2O_5 produced and generate phosphogypsum, considered a hazardous waste, at rates of four to five tons per ton of P_2O_5.

According to Elgafi, the process can save at least $300/ton of P_2O_5 produced, as well as five tons of water with improved energy efficiency. — *Oct. 19, 3:45 PM*

Hydrogen peroxide synthesis using a microreactor

Scientists at Japan's National Institute of Advanced Industrial Science and Technology (AIST; Tokyo, Japan; www.aist.go.jp) have developed a glass microreactor that allows smaller-scale production of hydrogen peroxide from hydrogen and oxygen.

Designed to provide a method for producing H_2O_2 at the point of use, the microreactor ensures the proper distribution of gas reactants and reaction solution over the catalysts. The reactor is fabricated using chemical isotropic etching with hydrofluoric acid, and also numerically controlled micromachining. Together, the two methods are able to generate narrow channels of width 0.05 mm, as well as wide channels in the sub-mm size range to control gas-liquid distribution over the catalyst and promote productivity.

The new microreactor has been used to generate aqueous hydrogen peroxide safely and at more than 5% (wt). — *Oct. 19, noon*

Realtime analysis of equipment gives full-system picture

A new methodology for realtime analysis of multiple pieces of equipment is offered by a division of Flowserve Corp. (Fairfield, N.J.; www.flowserve.com).

The approach and associated instrumentation can monitor multiple pieces of equipment, collecting high-resolution data that allow engineers to examine cause-and-effect interactions among all the components over the course of normal operational swings. As an example, Flowserve points to a case where the analysis approach monitored in realtime a complete feedwater system with ten locations for flow and six to eight pumps in operation, along with many valves and tanks.

Flowserve Integrated Solutions Group co-founder Dale Winterhoff says, "From this, you can get a true system picture relating cause and effect to operational performance."

The monitoring system generates "high-resolution data" that are programmable to be preprocessed according to engineers' needs. It has been employed in several industry sectors. — *Oct. 20, 3:30 PM* ■

Scott Jenkins

weftec® 2010

the water quality event™

The Water Environment Federation (WEF; Alexandria, Va.; www.wef.org) will host the 83rd annual Weftec technical exhibition and conference October 2–6 at the Morial Convention Center in New Orleans. The largest water-quality conference in North America, Weftec 2010 features 112 technical sessions, along with 35 workshops and six local facility tours. Its technical program and exhibition are designed to provide engineers and water quality professionals with the latest industry information and a host of educational and networking opportunities. The event will feature a keynote address by economics journalist Steven Solomon, author of the book "Water: the Epic Struggle for Wealth, Power and Civilization." The following is a small sampling of products on display at Weftec 2010.

This process converts sludge into biogas and fertilizer

This firm has built 20 thermal hydrolysis plants to date, with five plants now under construction. The firm also received an order for a sludge-treatment plant in Drammen, Norway, that will treat approximately 20,000 ton/yr of municipal sludge. Biogas generated at this plant will have an energy content of 16 GW/yr, suitable for cogeneration or as vehicle fuel. The digested sludge is dewatered into a pasteurized fertilizer. The sludge-treatment plant is scheduled for completion in the second half of 2011. Booth 7631 — *Cambi AS, Asker, Norway*

www.cambi.no

Measure flowrate in partially filled pipes with this device

The Tidalflux 4300 measures the volumetric flow of electrically conductive liquids in partially filled pipelines. The device's capacitive flow-level measurement system is built into the wall of the tube and works for levels between 10 and 100% of the pipe cross-section. The Tidalflux provides errors of less than 1% of the measured value, even in cases of rough product surfaces and distorted flow profiles. Booth 1967 — *Krohne Inc., Peabody, Mass.*

www.krohne.com

Moyno

These grinders ensure uniform sludge streams

Annihilator grinders (photo) are twin-shaft, solids-reduction units designed to ensure that harsh solids in primary sludge streams are ground uniformly and efficiently. The grinders can reduce the need for repairs by protecting downstream equipment from being plugged or damaged by large solids in a variety of industrial and municipal applications. Annihilator grinders feature optimized cutter combinations and a one-piece flange housing. The design places the mechanical seal and the bearing on separate stainless-steel sleeves to allow for quick onsite replacement of worn components without purchasing complete cartridge designs. Booth 5739 — *Moyno Inc., Springfield, Ohio*

www.moyno.com

An actuator that can adjust automatically to voltage

The new series EBVA electrical actuator (photo) automatically adjusts to the voltage provided within its standard voltage range of 85 to 240 V alternating or direct current. Also offered in a 12 to 24 V version, the actuator can generate torques of 177 in.-lb with an 11 s cycle time. The actuator is designed to work with a plasticizer-free ball valve. Booth 6646 — *Plast-O-Matic Valves Inc., Cedar Grove, N.J.*

www.plastomatic.com

Plast-O-Matic Valves

Fluid Metering

Inject hypochlorite for treatment of municipal drinking water

The Chloritrol valveless metering system (photo) can accurately measure, liquid sodium hypochlorite and calcium hypochlorite for treating municipal drinking water. Metering sodium hypochlorite presents a unique challenge because of the fluid's tendency to outgas. The Chloritrol System is a valveless, duplex-pump design where the primary pump injects liquid hypochlorite directly into the water main, and the secondary pump removes outgas bubbles from the system, preventing loss of pump-prime. The Chloritrol will self-prime against pressures up to 125 psi. Internal components are made of hard ceramic materials, which provide longterm, drift-free accuracy. In addition to sanitizing drinking water, the Chloritrol can be used to add sodium hypochlorite to municipal swimming pools, water parks, food-processing plants and in waste treatment applications. Booth 1561 — *Fluid Metering Inc., Syosset, N.Y.*

www.fmipump.com ■

Scott Jenkins

Note: For more information, circle the 3-digit number on p. 62, or use the website designation.

Fractionation Column

Pushing capacity limits

Starting around 1970, many new contacting devices became available for use in distillation, absorption and stripping columns. Those devices included high-performance random packings, structured packings, high-capacity structured packings, augmented crossflow trays, counterflow trays and cocurrent flow trays. At the request of FRI's membership, many of those devices were tested in FRI's 4-ft and 8-ft test columns.

The FRI test columns, and all of the ancillary equipment, were designed in 1952. The original designers of the unit imagined that the columns would contain simple single-pass, crossflow trays at 24-in. spacings. None of those designers imagined the eventual existence of today's two-pass, cocurrent flow trays, which have at least 50% more capacity. None imagined cocurrent flow trays at all. Nevertheless, subsequent to 2004 the following three cocurrent flow trays were tested at FRI: Shell ConSep, Koch-Glitsch Ultra-Frac and UOP SimulFlow. All worked well, but, it was beginning to concern the FRI membership that the capacities of the contacting devices were near to exceeding the capacities of the FRI test unit.

In 2007, FRI's Executive Committee, authorized the use of $2 million to upgrade the test unit and perform maintenance work that would extend its life for an additional 20 years. The first thing that FRI did was contract with FTS Inspection & Engineering Services (www.ftsinspection.com; Bartlesville, Okla.) to perform a mechanical audit of all vessels and lines. FTS ultimately concluded that even after 50 years of operation, the unit was mechanically sound and safe.

Thereafter, FRI staff engineers attempted to identify hydraulic and heat transfer bottlenecks in the unit. Project money was spent using the motto "One safe and correct step at a time". Over the last two years, many small and medium-sized projects were performed, with an overall target of 25% increase in unit capacity. The two biggest unit bottlenecks proved to be the steam-generating boiler and the cooling tower. Even repaired, the boiler's capacity was stuck at 17 million Btu/h. Meanwhile, the cooling tower was also stuck at 17 million Btu/hr and therefore needed to be refurbished. Temperature excursions in the 1990s had embrittled the plastic fill. Some areas of the fill were so under-irrigated that birds had built nests in between the fill sheets. The cooling tower was completely refurbished.

FRI's Technical Committee approved what was called an empty column test, which took place in October of 2009. The low-pressure column was emptied, so that no trays or packings would limit the unit's capacity. An auxiliary boiler was rented to run in parallel with the existing boiler. FRI's two reboilers were piped-up for parallel operation; so were the two condensers and the two reflux pumps. After about a week of lining out and minor troubleshooting, FRI's unit was run steadily at 24 million Btu/h — a 41% increase over the previous maximum.

FRI's maintenance budgets are annual and forever. FRI's upgrade project is now considered to be complete, at a total cost of only $716,000. FRI stands ready to test the next generations of trays and packings. Bring 'em on! ■

Mike Resetarits

Mike Resetarits is the technical director at FRI (resetarits@fri.org), a distillation research consortium headquartered in Stillwater, Okla. Each month, Mike shares his first-hand experience in this column.

CHEMICAL ENGINEERING
FACTS AT YOUR FINGERTIPS
Department Editor: Scott Jenkins

Controlling Membrane Fouling

The deposition and accumulation of suspended and dissolved particles on membrane surfaces leads to performance loss. Fouling can dramatically reduce the efficiency and economic benefits of a membrane process. The type of fouling and how strongly it appears depends on several parameters, including the following:

- Nature of solutes and solvents
- Membrane process
- Pore-size distribution
- Membrane surface characteristics and material of construction
- Hydrodynamics of the membrane module
- Process conditions

COMMON FOULING MODES

Membrane fouling can be a confounding problem in water treatment systems. Several of the most common types of fouling are shown in the following table.

FOULING EXAMPLES	
Foulants	**Fouling mode**
Large suspended particles	Particles present in the original feed or developed in the process can block module channels
Small colloidal particles	Colloidal particles can raise a fouling layer or block the porous structure of the membrane
Macro-molecules	Gel-like cake formation on top of the membrane or macromolecular fouling within the structure of porous membranes
Small molecules	Molecules such as substituted aromatics can adsorb onto the membrane structure and reduce the water content of the membrane, which lowers permeability
Scalants	Depending on the pH, salt may precipitate on the membrane. This reduces the membrane area and may reduce the water content in the membrane
Biological material	Growth of bacteria on the membrane surface, which leads to a gel-like cake on the membrane

REDUCING FOULING

Influence of the bulk solution

Properties of the bulk solution can affect membrane fouling, but whether these properties can be manipulated depends on the actual process conditions.

Adjusting pH, varying temperature and changing particle size are possible parameter changes that may be manipulated to influence the fouling behavior of a bulk solution. For example, operation at low pH can help prevent scaling of calcium sulfate by changing the solubility of the salts. This can significantly reduce the precipitation of calcium sulfate on the membrane.

Concentration polarization

Concentration polarization is the accumulation of rejected particles, especially during microfiltration and ultrafiltration, to the extent that transport to the membrane surface becomes limited. High flux through the membrane can cause rejected particles to accumulate on the surface of the membrane. Concentration polarization reduces the permeability of the solvent and can lead to a limiting flux, where an increase in pressure does not correspond to a rise in flux.

Reducing concentration polarization leads to higher limiting flux and lower fouling tendency. Controlling concentration polarization is essential for a process to be economically beneficial.

Concentration polarization can be controlled by increasing crossflow velocity or by increasing turbulence on the membrane surface, which increases the back transport of the particle away from the membrane. The challenge becomes balancing high fluxes and low fouling with low investment and operating costs.

Membrane properties

As an essential part of the membrane process, the membrane itself has a strong influence on fouling. Typically, hydrophilic membranes are specified because they exhibit an affinity for water, which is one of the main tools used to reduce the adsorption of foulants onto the membrane surface. A hydrophilic membrane is surrounded by water molecules, which work as a protective layer. The hydrophilicity and hydrophobicity of some polymeric membrane materials are shown in Table 2.

POLYMERIC MEMBRANE PROPERTIES	
Property	**Polymer**
Hydrophobic	Polytetrafluoroethylene (PTFE, Teflon) Polyvinylidenefluoride (PVDF) Polypropylene (PP) Polyethylene (PE)
Hydrophilic	Regenerated cellulose Cellulose ester Polycarbonate (PC) Polysulfone/polyethersulfone (PS/PES) Polyimide/polyetherimide (PI/PEI) (aliphatic) Polyamide (PA) Polyetheretherketone (PEEK) Cellulose triacetate (CTA)

Because hydrophilic membranes have lower chemical resistance than hydrophobic ones, their chemical stability and cleanability have to be evaluated as part of the selection process. It should be noted that most membranes are polymer blends.

Most polyethersulfone (PES) membranes contain some polyvinylpyrolidone (PVP) to increase hydrophilicity. The problem is that PVP is not stable against oxidizing agents, which may lead to changes in membrane porosity if not closely monitored.

Porosity

Porosity can reduce fouling during microfiltration and ultrafiltration. The strongest fouling is caused by the blocking of membrane pores. Therefore, their pore size should be smaller than the average particle size, and a membrane with a narrow pore-size distribution is preferred to avoid the blocking of bigger pores.

Influence of the permeate flux

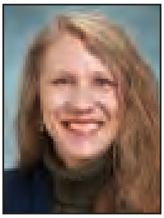
Critical flux is another factor that can be influenced to reduce fouling in microfiltration, ultrafiltration and nanofiltration processes. It is defined as the flux below which a decline in flux with time does not occur, while above this flux, fouling starts. At critical flux, the number of particles transported to the membrane is similar to the number of particles that diffuse away from the membrane. In operation below critical flux, the fluxes are reversible, which means that as long as flux is below the critical flux, membrane permeability is not changed by fouling. The advantages of critical flux are that constant fluxes and membrane properties can be sustained for longer periods. On the other hand, the conditions of critical flux require lower pressure, hence a higher membrane area is required. Therefore, the tradeoff between higher investment costs and lower fouling tendencies has to be assessed for each process.

The material in this column is adaped from the following article: Lipnizki, J., Strategies for Controlling Membrane Fouling, *Chem. Eng.*, September 2007, pp. 62–64.

WHO'S WHO


Pagenkemper


Roller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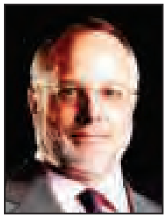
Muzyka

James Diamantis becomes director of strategic accounts for **Wayne Chemicals** (Fort Wayne, Ind.).

Bernhard Pagenkemper becomes sales director for the machinery div. of **Haver & Boecker** (Oelde, Germany).

Colfax Corp. (Richmond, Va.), a manufacturer of fluid-handling solutions, promotes *William Roller* to executive vice-president of Colfax Americas.

Kim Tutin is appointed to the newly created role of technology and innovation scout at **Georgia-Pacific Chemicals LLC** (Atlanta, Ga.).

Wolfgang Sautter becomes managing director at **Voith Turbo BHS Getriebe GmbH** (Sonthofen, Germany), a manufacturer of turbo gearboxes.

Darrin Drollinger is elected executive director of the **American Society of Agricultural and Biological Engineers** (St. Joseph, Mich.).

Hansen Technologies Corp. (Bolingbrook, Ill.) adds two regional sales managers: *Jim Walker* (northeastern U.S. and Canada) and *Jim Price* (southwestern U.S.).

DuPont (Wilmington, Del.) names *Doug Muzyka* chief science and technology officer. *Tony Su*, currently DuPont's director of marketing and sales in China, succeeds Muzyka as president of DuPont Greater China.

Ingen Biosciences appoints *Karine Mignon-Godefroy* as director of research and development. ■

Suzanne Shelley

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Applying CPI Temperature Sensors

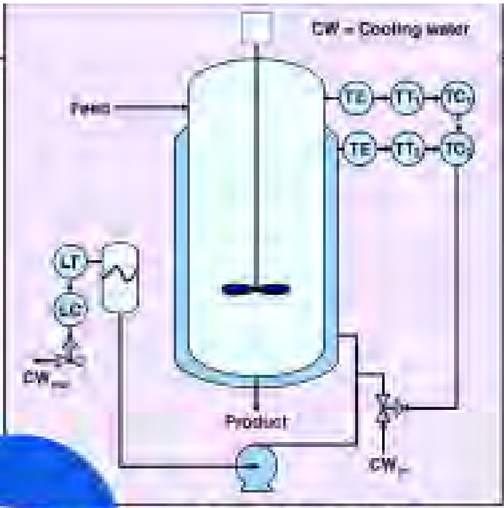


FIGURE 1. Typical P&ID diagram indicating TE, TT, and TC temperature-related points

Don't fall back on outdated rules of thumb that may no longer be valid, or 'plug and play' solutions that may not be applicable

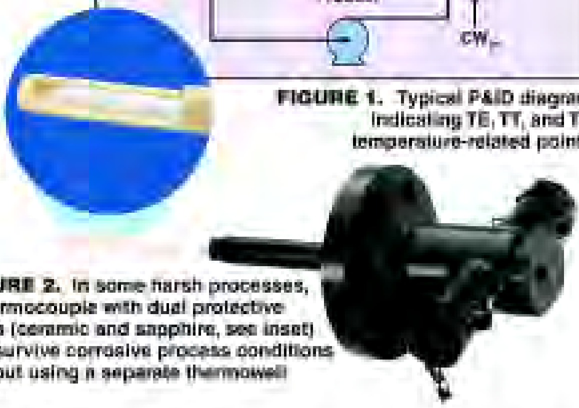


FIGURE 2. In some harsh processes, a thermocouple with dual protective tubes (ceramic and sapphire, see inset) can survive corrosive process conditions without using a separate thermowell

Khoi Nguyen
Emerson Process Management

Temperature is a critical measurement in the chemical process industries (CPI). If a measurement is not accurate or reliable, or the sensor is incorrectly chosen, it can cost a company thousands of dollars in wasted steam, extra feedstock or rejected product. Sometimes even a few degrees of error in a temperature measurement can be very expensive. Therefore, it pays to ensure that temperature measurements are reliable and accurate.

Selecting the correct sensor isn't just a "plug and play" procedure, where an engineer orders his or her favorite thermocouple (TC) and a 4-in. flanged thermowell for every 6-in. pipe. The process includes picking the correct thermocouple or resistance-temperature-detector (RTD) sensor, sizing the thermowell, deciding how and where to mount the sensor, choosing the type of transmitter to be used, specifying the method of transmitting the sensor signal back to the control room, and evaluating the kinds of diagnostic capabilities to be used.

What are you measuring?

It all starts with a piping and instrumentation (P&ID) diagram (Figure 1). Typically, all the P&ID tells you is where the process engineers determined the need for temperature elements (TE), temperature transmitters (TT) and temperature controllers (TC) on the batch reactor, process unit, distillation column, pipeline and so on. There might be a few dozen or a few hundred temperature sensors involved.

The P&ID does not tell you exactly where to install the temperature sensors, what they will be measuring, or any other details. It's the engineer's job to look through all the other process documentation and design practices, company standards and procedures, to determine the following:

- What fluid is being measured? Is it steam, gas, liquid or powder?
- What are the characteristics of the fluid? Is it corrosive, explosive or erosive?
- What is the temperature range?
- What measurement accuracy and response time are needed?
- What is the maximum flowrate?
- What is the maximum pressure?
- Are there any extenuating circumstances, such as vibration, inaccessibility, partially filled pipes, fault conditions, or steady-state versus transient conditions?

This information must be gathered for every temperature-sensing point. The characteristics of the fluid — such as being very corrosive — will determine the materials that must be used when specifying thermowells. The maximum flowrate and pressure, and the presence of high vibration, may also affect the choice of thermowell construction.

The temperature range will determine what sensor technology to use. The range covers everything from a cold startup to a possible over-temperature condition from a runaway reactor. If it is important to monitor a wide range, a different sensor will be required than that for a specific, narrow range. Some sensors and transmitters can be calibrated for higher accuracies at specific temperature ranges.

Some measurements in a process, such as tray temperatures, may have to be very accurate with a fast response to changing temperatures. On the other hand, monitoring the temperature of a storage tank may be acceptable with a less-accurate, slower-responding sensor.

In general, off-the-shelf temperature sensors and transmitters — if correctly sized and selected — can be used for 90% or more of process requirements. The remainder may require some extra care or specialty sensors.

MAKING NON-INTRUSIVE MEASUREMENTS

When thermowells cannot be used, temperature sensors can be clamped, bolted, welded or epoxied to a surface. One advantage of a surface temperature measurement is that it is easy to install; there's no tap required into the process—just clamp it on. The major disadvantages are a slower speed of response and less accuracy of the measurement.

For pipe clamp sensors, be sure to mount the transmitter the proper distance away from the sensor, so that heat from the pipe and ambient temperature does not affect the transmitter (this is called the transmitter temperature effect). Follow manufacturer recommendations on the proper distance to reduce the maximum-allowable rise in housing temperature.

FIGURE 3. Pipe clamp sensors are useful in applications where process intrusions are impractical or unreliable

For all surface sensors, be sure that the sensor itself is insulated from the ambient temperature. This can be accomplished by wrapping the sensor connection and pipe with insulation. Otherwise, the sensor may produce a false measurement. In addition to welding or gluing the sensor itself, allow the sensor wire to remain in contact with the surface for a short distance. ❑

FIGURE 4. A temperature transmitter is a rugged, field-mounted device that can be installed directly on the sensor and thermowell

Immersion vs Non-intrusive

To make the most-accurate temperature measurement, the sensor should be immersed so that it comes in direct contact with the fluid. Since many fluids in the CPI are corrosive or erosive and can destroy a bare sensor quickly, the most common way of making temperature measurements is to use a thermowell (see box, Installing Thermowells).

A thermowell is a hollow tube, often tapered, that projects into the fluid to protect an immersed sensor. Because it is immersed in the fluid, the thermowell is at the same temperature as the fluid. A temperature sensor, inserted into the thermowell, measures the temperature of the thermowell and, therefore, the fluid. A thermowell can be used on any pipe with a diameter larger than 1-1/4 in., or it can be inserted into the side of a tank.

Depending on the fluid, it may be suitable to immerse a temperature sensor without a separate thermowell. Some sensors are available with one or more protective layers, and can be immersed in certain fluids. For example, a producer of fertilizer and ammonia uses a gasifier to convert super-heavy waste oil into syngas. Process conditions are severe: Temperatures above 1,600°C, with a corrosive environment and toxic gases, causing premature failure of thermocouples. The plant switched to temperature sensors (Figure 2) that have an external, protective ceramic tube and a second, internal sapphire tube. The protective tubes doubled sensor life from 12 months to 24 months as well as the resultant benefits to throughput, uptime and maintenance costs.

If the measurement point is on a smaller pipe, or is in a process situation making a thermowell unfeasible, the engineer must consider a non-intrusive or "skin" temperature measurement (see box, Making Non-Intrusive Measurements).

Non-intrusive measurements are relatively easier to make, less expensive than thermowells, and can be used when it is not possible to use a thermowell. For example, an offshore oil producer was experiencing paraffin buildup in piping and needed to measure the temperature of the pipe. However, no pipe penetrations or welded connections, which might fail under high vibration conditions, could be used. To solve the problem, the producer installed pipe clamp RTDs in pipe locations where paraffin buildup was common, eliminating the need for pipe penetrations (Figure 3).

Note that surface measurements are not as accurate as immersion methods. Typically, a surface measurement is only accurate to 1%, while immersion measurements can be accurate to within 0.1%. This may pose a problem, depending on the process. For example, if the process involves a very expensive feedstock and needs to control its temperature with 0.1% accuracy, but the feed line is too small to install a thermowell, process engineers will have to be mobilized. Perhaps they can design a surge tank, elbow or a larger section of pipe that will allow a thermowell to be installed for better accuracy.

Thermocouple or RTD?

When all the characteristics noted above have been determined, it's time to choose between a thermocouple and a resistance temperature detector (RTD). While other sensors, such as thermistors and infrared, can be used, the vast majority of temperature sensors in the CPI are either thermocouples or RTDs.

Entire handbooks, articles and chapters in instrument books have been written describing the differences, advantages and disadvantages of each, so this discussion is limited to a brief summary. The reader is urged to research the subject in more detail when selecting a sensor. Vendors can help.

Thermocouples. A thermocouple sensor consists of two wires of different metals, that are joined at one end. The junction creates a voltage that varies with temperature. In comparison, an RTD changes its resistance with temperature.

Thermocouples can be relatively inexpensive and are available in various types that are calibrated to specific temperature ranges and accuracies. The four most popular types are J, K, E and T which cover temperatures from below 0°C to well above 1,000°C with accuracies of about 0.4%. Thermocouples can be ordered with various "sheaths" and junction types, which affect the speed of response, resistance to electrical noise, and resistance to chemicals, abrasion and vibration.

A thermocouple has to be connected to a transmitter or marshalling point via thermocouple wire, which is a "lead wire" that is made of the same material as the T/C. If the distance between the sensor and transmitter is too large, the signal can be affected by heat or electrical noise. One problem with T/Cs is their tendency to "drift" over time, requiring re-calibration in the field.

RTDs. An RTD is more accurate than a T/C. The IEC (International Electro-

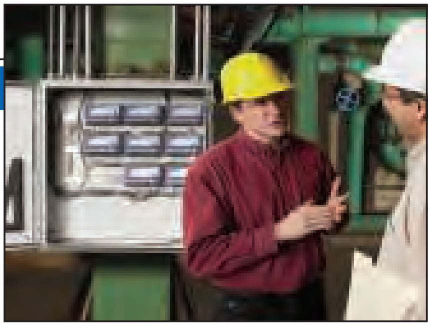


FIGURE 5. High density transmitters, such as these, can accept up to eight thermocouple or RTD inputs each, thus replacing many individual wire runs in applications such as columns and reactors

FIGURE 6. A wireless temperature transmitter can be easily installed in remote or inaccessible areas, where running wire would be too impractical

technical Committee) industry standard for platinum RTDs is 0.12%, making an RTD more than twice as accurate as a thermocouple. RTDs also have lead wires, but they can be configured in two-, three- or four-wire circuits. A two-wire lead circuit has problems with lead wire compensation and should only be used for short connections. The best solution is a four-wire circuit, which is used when a high degree of accuracy is required.

RTDs and thermocouples are available in various versions to suit virtually any industrial application. As a general rule, RTDs are more accurate and drift less but are more expensive than thermocouples. RTDs are useful up to more than 600°C, while thermocouples can go up to several thousand degrees Celsius.

Signal conversion, transmission

The millivolt signal from a thermocouple or the milliohm signal from an RTD must be converted to a form suitable for use by the control system. Typically, this function is performed by an analog-to-digital (A/D) converter obtained via one of the following:

- Wiring directly to an analog input card in a programmable controller (PC), programmable logic controller (PLC) or a distributed control system (DCS)
- A multiplexer that converts it to a digital value that can be transmitted over a network
- A temperature transmitter, which converts the sensor signal to a 4–20-mA signal that can be sent over long distances on a twisted pair of wires or wirelessly

Wiring the sensor signals directly to a PC, PLC or DCS analog-input card works only if the control device is nearby. The cost of running lead wires is extremely expensive, and long distances can be affected by electrical noise. The lead wires themselves act as antennas that pick up noise.

A multiplexer is typically mounted in a field enclosure centrally located among several sensors. A multiplexer might accommodate signals from dozens of nearby sensors, with each sensor attached to an analog input channel. Multiplexers, along with remote terminal units (RTUs), data acquisition systems and data concentrators are widely used, especially in rehabilitation or retrofit applications where it is too expensive to run additional sensor wire or twisted pairs through existing conduit or underground passages.

A temperature transmitter is an industrially hardened device that can be installed near the sensor, even in hazardous areas (Figure 4). Twisted-pair copper wires (or wireless communications) from each transmitter convert and carry the signal from the field to the controller. Using transmitters can be economically justified due to the lower cost of copper versus thermocouple lead wire and the improved reliability and performance of transmitters. The recent advent of so-called high-density transmitters combines the multiplexing technology (the ability to receive multiple sensor inputs) with reliable transmitter technology to provide an option that is both robust and effective from a cost-per-point basis in applications communicating data from large quantities of sensors. High-density transmitters complement traditional single-point transmitters to provide the plant designer with measurement communications options for different plant applications.

The choice of how to convert the signal depends on several factors:

- Distance to the converting device (long distances affect accuracy)
- Electrical noise in the area, which may interfere with lead wires
- The number of nearby sensors, which influences whether a high density transmitter or a multiplexer RTU can be used

In recent years, additional factors have entered the equation. CPI decision makers are now much more aware of the high cost of installing wires. Estimates for installing wires currently say that parts and labor add up to a cost of $50 to $200 per foot, and up to $2,000 per foot in hazardous areas. With such high costs, wiring sensor signals directly to a controller is rarely an option, unless the controller is in the immediate vicinity of the process unit.

Instead, chemical plants are turning to transmitter technology (analog, HART, fieldbuses and wireless) because these methods drastically reduce wiring costs while improving measurement reliability.

A high-density transmitter can save a project a considerable amount of money, because it replaces up to eight individual transmitters with a single device. For example, a glass manufacturer re-instrumented 12 vapor-deposition furnaces. Each furnace had 16 temperature monitoring points, so using conventional transmitters would require 192 devices. Instead, they installed high-density, eight-input transmitters (Figure 5), which accept eight sensor signals. The savings amounted to $10,000 in transmitter costs, and $21,000 in labor costs. The labor savings associated with the installation arose from reduced wiring and terminations, as well as the transmitter's ability to detect when a thermocouple fails or needs to be recalibrated, eliminating unnecessary maintenance.

Improved transmitters

Another factor entering the equation is the increased capability of transmitters. While they were once limited to making the 4–20-mA conversion, transmitters now perform a variety of functions that make them more attractive and cost effective.

Not too many years ago, a transmitter was considered an expensive

INSTALLING THERMOWELLS

When measuring the temperature of liquids or gases in a pipe or tank, the sensor should be immersed into the fluid. In many cases, this is accomplished by installing a thermowell — a hollow tube — into the pipe or tank so that it comes into contact with the fluid to be measured. An immersion probe containing a temperature sensor is then inserted into the thermowell from the outside so that the sensor fits flush against the end of the thermowell. Because the thermowell is immersed in the process fluid, it transfers the temperature of the fluid to the sensor.

Thermowells can be manufactured from a variety of materials (predominantly metallic but also ceramics and polymers), specifically to survive the erosive or corrosive effects of the process fluid and to protect the sensor. The thermowell manufacturer will recommend the size, type (welded or machined) and material (304 SS, 316 SS, brass, and so on) for each application.

Some general rules

Thermowells can be used in pipe diameters as small as 1-1/4 in. In small pipe sizes, be sure the thermowell does not become an obstruction to flow.

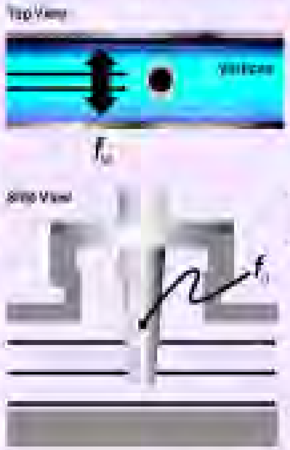


FIGURE 7. Incorrectly designed and installed thermowells may fail at high flowrates. Once the wake vortex-induced force (at frequency f_w) approaches the thermowell's natural frequency (f_n) catastrophic failure can occur unless correct thermowell calculations are performed

- Install the thermowell 3–5 pipe diameters away from elbows, flowmeters or other devices
- Follow the manufacturer's recommendations on pressure, temperature and fluid velocity. Thermowells can fail under certain conditions including shedding vortices that lead to excessive vibration and catastrophic failure. Make the calculations necessary to select the correct thermowell (Figure 7)
- If the pipe will only be partially filled, be sure the bottom of the thermowell is immersed in the process fluid

A new thermowell-calculation industry standard — ASME PTC19.3TW — will be coming out this year to help you make the calculations necessary to select and size thermowells. This standard was created by an industry-wide coalition of end users, vendors and technical institutions. ❑

luxury that could only be justified by the need for signal loop integrity and reliability. Everything else was wired directly to the control room. Today, the cost of wiring a signal to the controller rivals or exceeds the cost of a transmitter. Transmitters can be mounted directly on the thermowell or pipe clamp, next to the measurement point on a pipe stand or bracket, or as DIN-rail devices in an enclosure.

A modern transmitter has many attractive features, not the least of which is its diagnostic capabilities. Built-in intelligence allows a modern transmitter to perform diagnostics, detect thermocouple degradation, signal conditioning and loop tests, and correct for the nonlinearity of RTD sensors. For example, temperature sensors drift and burn out over time. If a sensor fails, the transmitter's diagnostics can detect the failure and inform the control room. A transmitter can deal with sensor drift by using two sensor elements; if the difference between the two measurements is significant, the transmitter will determine that drift has occurred.

A transmitter with two elements can also serve as a so-called hot backup: That is, if one sensor fails, it uses the other one to provide enhanced measurement reliability.

Finally, some transmitters may eliminate the need to install a temperature sensor in certain places. For example, Coriolis flowmeters often measure the fluid temperature so they can calculate viscosity, concentration and other parameters. Check with other instrument engineers on your project: You may find they have specified similar transmitters, and you can eliminate these temperature sensing points by simply taking the temperature signal from a flowmeter.

The rising tide of wireless

Wireless temperature transmitters are becoming much more popular in the CPI for two major reasons:

- They drastically reduce the cost of wiring. A wireless transmitter can be battery powered, solar powered or use a local power source. This allows a plant to make temperature measurements in locations, such as a remote storage tank, where communications wiring costs would otherwise make installation too expensive
- Wireless transmitters (single-point or high-density) can be installed virtually anywhere (Figure 6), quickly and with minimal labor costs. With this versatility, a temperature sensor can easily be added to an existing system to make temporary measurements, experiment with sensor placement or handle last-minute requests

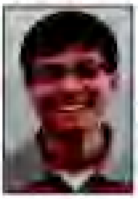
A wireless temperature transmitter that sends data via the globally adopted WirelessHART standard easily interfaces into any modern control system. A WirelessHART network comprised of temperature, pressure, level, flow and other devices provides plant designers and end users with the optimal combination of instrument flexibility, reliability, expandability and ease of use required for modern CPI plants.

Approaching the task

At one time, specifying and installing temperature sensors was a fairly simple task: Just select the proper thermocouple or RTD and wire it back to the controller. The task is much more nuanced and complex today. Engineers should be careful to not fall back on rules of thumb that may no longer be valid, and so-called "plug and play" solutions that may not be applicable. Specifying the correct sensor, installing it properly and bringing the signal back to the control room requires much more analysis than just picking a thermocouple or RTD out of a vendor's catalog. However, the extra analysis will pay off with a solution that is accurate, reliable and cost effective. ■

Edited by Rebekkah Marshall

Author



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Polymer-Based Piping Systems in the CPI

If properly used, polymeric piping and components can be cost-effective alternatives to those made of traditional materials

FIGURE 1. Polymer-based piping systems deliver many advantages, such as those listed here

Stephan Schüßler
Georg Fischer DEKA GmbH

Polymeric piping systems have been used in well-defined segments of the chemical process industries (CPI) for decades. They have proven their sustainability to manage almost any chemical load up to temperatures of 100°C in the moderate pressure region ($p < 10$ bar) if the choice of the respective piping-system material has been made correctly. However, their potential of exceptional performance is still not fully reflected by the number of polymer-based piping systems used in the CPI.

This article gives a comprehensive review of the advantages and limitations of polymer-based piping systems in the CPI. By also quoting representative case studies as well as outlining trends, aspects for further discussions for this topic are given without devaluating possible non-polymeric material alternatives.

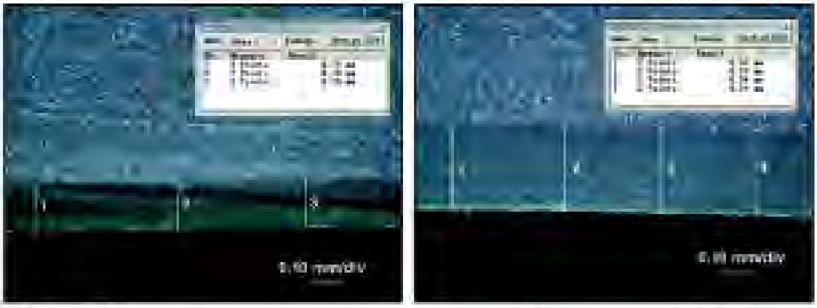

FIGURE 2. These closeups (transmission mode; 1:200) of microtome cuts of PVC-U piping-system components after six years of service (37 wt.% hydrochloric acid) demonstrate the importance of polymer formulation. The image at the left is for premium industrial grade pipe, while that on the right is for a standard fitting; differences of 100% in the corrosion attack are evident in the non-premium PVC-U

State of the art

Piping systems become relevant where (mostly) liquid or gaseous media are conveyed, distributed, dosed or stored. Starting with their introduction about 50 years ago, polymer-based piping systems have been fully established in many segments, such as building technology, gas and water supply [*1*]. However, in many industrial sectors of the CPI their application requires a much more distinct consideration, as the transported media are mainly chemicals that are conveyed above ground and combined with a well-defined set of operation conditions regarding, for example, pressure, temperature and concentrations of media. Based on this, each application in the various CPI segments can be looked at as being a unique and sometimes quite-challenging case. Projects in the CPI typically require one or more of the following constraints for a piping system [*2*]:

- Considerable safety margins or dry-running properties as a system
- Sufficient chemical resistance, or predictable or manageable corrosion
- Uniform corrosion behavior of all components with direct media contact
- Simple maintenance, extension or restructuring of the system
- Attractive price/performance ratio
- High level of availability of the components
- Availability of a steady growing pool of longterm field references
- Ease of installation

Compared with other industries, the application of pipe usage in the chemical sector is characterized according to a demanding standard involving safety and an enormous knowledge of actual field experience gained with a given material in the specific process of interest.

This might account for why polymers remain a comparatively small part — about 10% — of the global CPI piping market [*3*]. However, looking at this from the perspective of a material scientist who is able to judge the technical potential of the polymeric materials in such a field of application, different and higher shares should be expected and the rate of adoption is increasing.

Advantages & characterization

Chemical resistance. The most decisive aspect for piping materials in the chemical sector is long-term chemical resistance. It is rather self explanatory that the chemical resistance profile of

FIGURE 3. In addition to piping, some vendors also offer a comprehensive line of components. Those shown here are for a PP pressure-piping system

the respective polymer-based piping-system component will be strongly defined by its chemical identity. However, for a non-specialist it is not at all straight forward to accept that different polymers show very large differences in their chemical resistance profiles. Additionally, this profile can be influenced, shaped and adapted to a specific application when the impact of the polymer formulation is fully exploited. Furthermore, the enormous influence on the polymer properties and the chemical resistance can also be gained by the processing of the formulation. In some cases both aspects can be of similar impact. Figure 2 shows microtome cuts of unplasticized, polyvinyl chloride (PVC-U) piping components used in a typical CPI application after several years of continuous service. This simple example illustrates the range of effects of chemical resistance linked to the formulation of the polymer.

Cost-to-performance ratio and availability of the system components. When comparing polymer-based piping systems to alternative metallic systems, a number of attractive features of the former becomes evident: Low weight, thermal and electrical insulating properties, often simpler jointing techniques, as well as easier handling during installation. Additional synergies regarding project pricing are possible by exploiting the ability of some manufacturers of piping system components to offer complete and harmonized systems encompassing a wide range of dimensions and pressure ratings. Such systems also cover integrated solutions of measurement and control units as well as various valve systems. This synergistic aspect can even be further enlarged in cases where the manufacturer is able to supply entire piping systems for different applications along with engineering or consulting support to the project. Figure 3 shows a good example of such a system fabricated out of polypropylene (PP).

As a rule, such standard systems can always be adapted to individual requirements (for example, regarding the dimensions) by special customizing of units for the processors. This in turn is appreciated by the CPI-users, and has always been a strong argument for the use of polymers in CPI piping.

The various components of such systems are usually backed by a powerful logistic system that enables global availability. In general, they are globally compatible to the respective products of different vendors as long as imperial and metric sizes are not mixed.

Fabrication is usually based on the respective standards (ISO EN, DIN or ASTM), and within a given system you may find a harmonization of geometrical tolerances. Established manufacturers also take care that special approvals will cover the entire range of relevant system components. For further reading in this field, see Ref. 4 on planning fundamentals.

Multiple jointing

Jointing of polymer-based-piping system components is quite versatile and subjected to the choice of the polymer. Established approaches from the draining, sewage and utility segment, such as removable socket joints (push-fit-fitting), clamping or using electro fusion are not utilized in chemical, petrochemical and other sectors of the CPI. In these application areas, thermal welding and solvent jointing are predominant; in case of removable and multiple jointing (for example, prefabricated spools), the use of flanges has become state of the art — especially for jointing of spools made out of different materials.

Welding. The welding of polymer piping in the CPI is split into socket-, butt-, fusion- and rod-welding, where the former two approaches are used for pure thermoplastic-pipe welding, and the latter two are established in the field of fiber-reinforced thermoplastic piping (so-called dual laminates).

For butt-fusion methods there is a choice between simple contact (hot plate) and non-contact (infrared [IR]) welding. Using IR welding has the advantage (among others) of a decreased formation of welding beads due to the reduced volume of the polymer being heated. A "beadless" weld can be achieved by the so-called bead-and crevice-free (BCF) welding process that has widely been accepted as a jointing standard in the pharmaceutical and semiconductor industries (see Figure 4).

Cementing. For solvent-cement jointing, various solvent-based glues are used; the respective formulations are fine-tuned to match the individual requirements defined by the polymer and the field of application. It is worth noting that this technique has also reached the state that jointing of PVC-U, for instance, can be done with a special solvent cement that is even able to cope with long-term and steady contact to strong oxidizers, such as concentrated sulfuric acid.

As soon as the dimensions of fabrication tolerances of pipes and fittings exceed a certain limit, solvent-cement jointing becomes no longer practically feasible due to both the gaps that need to be "bridged" and the procedure of assembly (required forces, application of the cement and so on). In all those cases the dual laminates (based on welding and flanging) are then widely accepted as an alternative. Note that no solvent-cement jointing is recommended in the field of dual laminates.

TABLE 1. THE VERSATILITY OF POLYMER-PIPING SYSTEMS

Concept	Jointing technology	Typical OD-range / mm	Characteristics
Thermoplastic pressure pipe	(Butt-fusion) welding; solvent cement jointing, flanging	16 – 2,000	Standard in the CPI
Double containment	Solvent cement jointing; butt-fusion welding	20 – 225 (Inner pipe)	Increased safety due to pipe-in-pipe concept; outer shell frequently made of transparent PVC-U
Dual laminates	Welding (butt fusion / rod); flanging	16 – 2,000	Increased safety due to pipe-in-pipe concept; significant increase of the range of applicability as the thermoplastic inliner is only used as the "chemical barrier"; all mechanical load is handled by the external FRP-layer
Tubes	Push-fit sockets, flaring, screwing, customized solutions	< 2 in.	Coiled tubing made, for example, from full fluorinated polymers used in double containment piping are of particular interest for very demanding CPI-applications (for instance, electronic-grade chemical media handling)
Ventilation pipes	Socket jointing in combination with solvent cement joining or (rod-)welding	< 800	Frequently the only economical alternative to handle corrosive exhaust fumes
Customized piping	Multiple and very individual	Usually < 250	Pipes as semi-finished products; mostly no system-concept required

QA and standardization

Since the market introduction of polymer-piping systems a few decades ago, many national standards (for instance, DIN in Germany or ASTM in the U.S.) have been elaborated, continuously developed and stepwise accepted — also globally in many cases. Nevertheless, standardization of polymer-piping systems is completed by international application norms which are updated regularly by international competent working groups. For selected applications (such as drinking water or gas supply) additional approvals combined with external third-party testing supervised by a separate quality assurance body (for example, the DIN Certco in Germany) do effectively increase the quality level of such approved piping systems. As a rule, such approvals are based on even stricter national quality-assurance (QA) regulations (such as various DIN Certco guiding rules). For the global CPI it is very important to consider that the so- called German Institute for Building technology (DIBT; Berlin; www.dibt.de) provides a unique, worldwide approval based on such QA guidelines. This DIBT approval is the basis for a very narrow-meshed testing program and documentation of each lot of piping products. In particular, while setting a strong priority on the commitment to a clearly defined material formulation — to be disclosed to the DIBT by a non-disclosure agreement (NDA) and an integrated third-party testing procedure — approved products are also linked to a comprehensive chemical-resistance list that will also provide additional safety factors (distinguishing between temperature and media concentration) for appropriate dimensioning of the piping system. Thus, this DIBT-approval should be considered as much more than just a national German QA issue; it is rather a very helpful specification tool for end-userss making use of polymer-based piping systems for conveyance of hazardous chemical media.

In addition to its presence in the standards of the American Welding society (AWS), the jointing of such systems is documented and updated in comprehensive detail by the guidelines issued by the German Welding Society (DVS) [*5*]. Similar to the fabrication standards of polymer-piping system components, these guidelines have been globally accepted over many years, laid down in many end-user specifications and are continuously being updated and completed. Furthermore, the regular training and education of polymer-welders is based on these standards. Note that a given DIBT pipe approval does directly refer to the respective DVS guidelines regarding planning, construction and joining of the respective piping products. Thereby, additional support is given to keeping up with quality.

In addition, many competent end-users (typically bigger companies in the CPI) rely on the use of a sensible and in-house developed system of standards, which are also part of their respective process specifications. Of course, these internal and individual standards are also regularly reviewed and updated based on a steady growing experience gained from practical long-term use of polymer piping systems.

Versatile piping concept

There is no other class of material showing a larger variety of piping concepts available for CPI applications than polymer pipes. Table 1 presents a number of these.

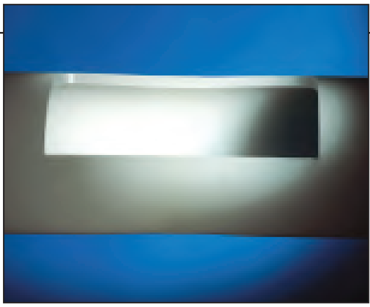

FIGURE 4. Shown here is view of a BCF weld of PVDF (polyvinylidene fluoride). The energy for melting the polymer is introduced from the outside; the formation of a crevice-free and smooth inner surface in the joining region is achieved by temporarily inserting a balloon filled with compressed air

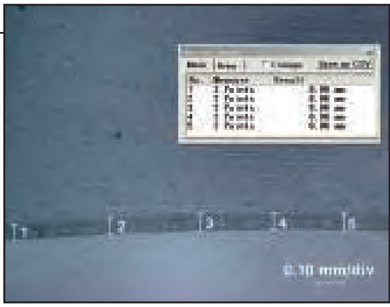

FIGURE 5. After 17 years of continuous contact with 35 wt.% hydrogen peroxide (25°C), only 60 μm of corrosion attack is measured with PVC-U piping components. Such minor traces of corrosion do not influence the function or safety of the system at all

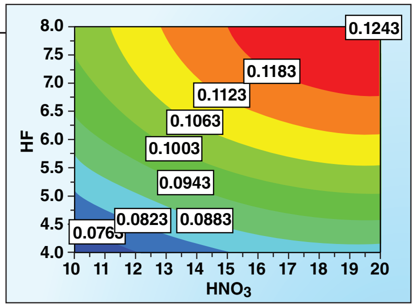

FIGURE 6. This graph show the isothermal diffusion coefficients of mixed acid (HNO_3/HF; T= 65°C) with variable concentration (wt.%) in PVC-U determined from the combination of laboratory-scale immersion testing and simulations (source: Swerea KIMAB [7])

A good investment over decades

Although polymer-based piping systems have not reached the status of metal-based piping in CPI usage, there are a variety of quite demanding segments in CPI where the specific advantages of polymer pipes have been understood, systematically and successfully exploited for many years now [6]. The global chlorine industry, basic-chemical synthesis, logistic partners for chemical media, surface technologies (for example, galvanizing and pickling industry), pulp and paper, as well as power plants are the most important fields of application. The combination of the practical experience gained from these sectors and the knowledge derived from other fields of application leads to a considerable pool of data supporting the assessment and further understanding of the longterm performance as well as to assist in the decision making for material selection. The continuous evolution of that knowledge-pool is efficiently supported by both an active international network of polymer-corrosion specialists as well as international and national joint efforts in polymer-corrosion research of institutes, universities and industry-partners. In this concern the immense contribution (30 years of active research) of the Swerea KIMAB (former Swedish Corrosion Institute, SE) as well as the recent activities of the NACE (U.S.), Eurocorr (EU), FGK/GfKorr and KRV (Germany) should be recognized. The result of this work is primarily a steadily increasing pool of longterm-field references, thoroughly evaluated case studies of key applications as well as the buildup of systematic databases to extrapolate or forecast the corrosion process of a given polymer in contact with chemical media under varying service conditions. Figures 5 and 6 provide an impressive example of the status of such efforts in this field.

Physical and natural barriers

A comparatively high thermal expansion, longterm creeping under mechanical load as well as a significant reduction of mechanical properties under increasing temperature are the well-known, inherent shortcomings of polymers that have to be considered in any polymer-product application. Those processes are, of course, well-understood, and corresponding material data are always considered in the material selection, design and dimensioning. Polymer-specific safety factors and very reliable data to quantify the longterm creep behavior support this selection process. When using smaller pipe-dimensions (O.D. typically < 110 mm) in the CPI, many experienced end-users specify the application of thick-walled piping systems for safety reasons (for instance, pipe series 9 for PVC-U). As already mentioned, the use of dual laminates for handling of chemical media at elevated temperatures or higher pressure (or both) is also a widely applied tool to fully exploit the potential of chemical resistance properties of a given polymer and to surmount some of the polymer-inherent shortcomings. Keeping a proper bonding of the inliner to the FRP sleeve maintained as well as not exceeding the critical wall-thickness of the inliner are necessary preconditions to guarantee a safe operation.

The energy-transporting properties of polymers can strongly be influenced by compounding with suitable functionalizing additives; by doing so, even an increase of the thermal conductivity of a few orders of magnitude under retention of the electrical insulation-properties is possible.

The non-destructive monitoring of corrosion processes in polymer piping is still an unfulfilled demand of many end-users and the realization of such a concept is currently not in sight. However, efficient management of corrosion knowledge, practical experience, a rational understanding of the chemical processes and materials involved can already give a good and reliable basis for a sustainable and safe operation. Competent manufacturers are always good partners for the end-customer in case of need.

Quo vadis industrial piping?

Despite being a small branch of the industry, industrial piping and piping for the CPI have always been very innovative. During the last few years, the impulse for innovation in the chemical industry shows a focus at the site of compounder and processors rather than at the site of the raw material suppliers. In the field of materials and piping concepts, a trend towards more pressure-resistant and stiffer pipes has become evident. The various approaches are quite versatile and cover innovations such as layered structures, continuous welding of oriented polyolefin tapes on polyolefin pipes made from the same polymer; and the use of innovative copolymers or fillers, to name just a few. The topics "functional layers" and "nano-technology" have just emerged in practically relevant concepts for this type of industry and are expected to strongly influence the innovation in this field in the course of the coming years.

With regard to the jointing of polymer piping, the butt-fusion welding of PVC-U and C-PVC (post-chlorinated PVC) is currently established as an alternative to the conventional solvent-cement jointing of these types of materials.

Concluding remarks

Polymer-based piping systems have been used with great success in the CPI for many years now. The various systems that are currently available in the market are capable of covering practically any CPI application involving temperatures up to 100°C and pressures to 10 bar. In larger projects, tremendous advantages in cost versus performance can be exploited when fully considering that various polymer-based piping systems for different applications within a given project may be available from one manufacturer.

A well-developed standardization effort coupled with an effective QA of the products and an individually elaborated specification by the end-user will contribute to further growth of the use of polymer based piping systems in the CPI. Innovation and a steadily growing understanding of the supply chain regarding longterm performance and corrosion details of polymer pipes are expected to support this process. ■

Edited by Gerald Ondrey

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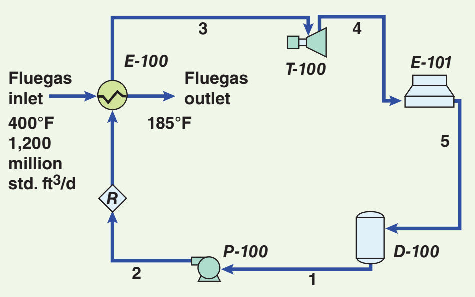
Recover Waste Heat From Fluegas

Adding an organic Rankine cycle system to generate power onsite can help operators optimize the overall economics of combustion-related systems and emissions controls



Working fluid: R-245fa

Turbine (*T-100*) generation, hp	1.291 x 10^4
Condenser (*E-101*) consumption, hp	57.72
Pump (*P-100*) consumption, hp	1,031
Total power recovery, hp	1.182 x 10^4
Total power recovery, kW	8,815

FIGURE 1. This simulation flowsheet for an ORC system shows the basic equipment, flow streams and power requirement and recovery based on R-245fa as the working fluid. The stream numbers indicate the order in which the streams flow

Ali Bourji, **John Barnhart**,
Jimmy Winningham, **Alan Winstead**
WorleyParsons

The ever-increasing cost of fuel and relentless push for environmental responsibility are constant factors in the profitability of many chemical process operations, and fluctuations in the prices of natural gas and other fuels can make it hard to predict future energy costs and the impact of such fluctuations on profits. However, the impact of fluctuating fuel costs, and the regulatory uncertainty related to managing emissions can be reduced by increasing energy efficiency and incorporating innovation into the design of any combustion-related system that produces fluegas.

Numerous industrial processes involving furnaces, heaters, kilns and boilers that combust fossil fuels release large quantities of energy in the form of waste heat that is contained in the fluegas. An organic Rankine cycle (ORC) system can efficiently utilize this waste heat to generate electricity, even from relatively low-temperature fluegas streams.

An ORC resembles a typical Rankine cycle, but instead of circulating water as the working fluid, an ORC uses a refrigerant — typically an organic fluid such as ethane, propane, propylene or various name-brand refrigerants, such as R-245fa (discussed below). An ORC also operates at lower temperatures compared to the more widely used steam-based Rankine systems. For instance, steam-based systems typically operate at temperatures corresponding to low-pressure steam; that is 250°F or higher. By comparison, an ORC can efficiently operate at temperatures below the boiling point of water, from 212°F to as low a temperature as desired for a given application. Thus, the use of an ORC can be a more cost-effective method for capturing waste heat from fluegas compared to the use of a traditional steam-based Rankine cycle. This is accomplished by reducing fluegas temperatures below what is possible in a steam-based system without the need for a complex heat exchange system.

When properly designed, an ORC system can remove more heat from a fluegas stream than a steam-based Rankine system and thus can be effectively used with cooler fluegas streams compared to those produced by steam-based Rankine systems.

Meawhile, most fluegas-treatment methods, such as those involving fluegas scrubbing, carbon capture, and carbon sequestration require the fluegas stream to be cooled prior to its introduction into the treatment train. Thus, the addition of an ORC system can be of great benefit when used in combination with a downstream fluegas-treatment system, and can help to improve the overall economics of fluegas treatment by generating additional power from waste heat in the fluegas stream — heat that would otherwise simply be lost to cooling water or to the atmosphere.

The basic ORC scheme

The simple process flow diagram that is provided in Figure 1 shows a basic ORC system. The liquid refrigerant (also called the working fluid) flows from the surge drum (*D-100*) to the pump (*P-100*) where the fluid is pressurized. The pressurized fluid is then sent to the evaporator (*E-100*). The evaporator vaporizes the working fluid by heat exchange with the fluegas stream. Once vaporized, the working fluid then enters the turbo-expander/generator (*T-100*) where, by process of expansion, the fluid produces usable work in the form of electrical energy. After expansion, the fluid enters the air-cooled condenser (*E-101*) where it is condensed back to liquid form and returned to the surge drum.

Figure 1 also provides values for the basic fluegas stream variables along with a small table that shows the quantity of power produced by the turbo-expander/generator train, the components that are power consumers (and their respective power consumption), and the net power recovered by the system.

Power-recovery rates will vary for different applications based on fuel

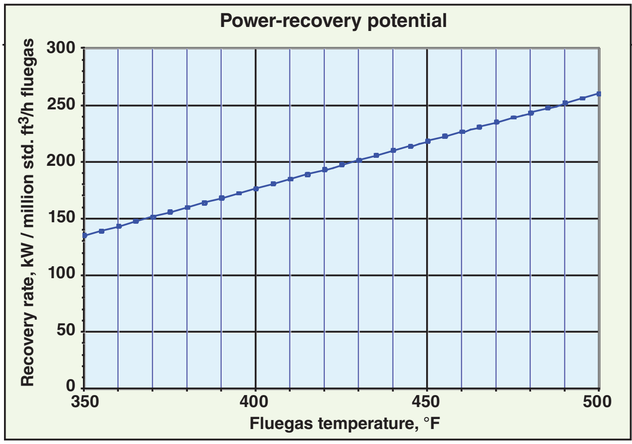


FIGURE 2. This chart, showing the power-recovery potential at various fluegas temperatures, can be used to approximate the power-recovery rate for various fluegas inlet temperatures at a given ambient temperature of 70°F

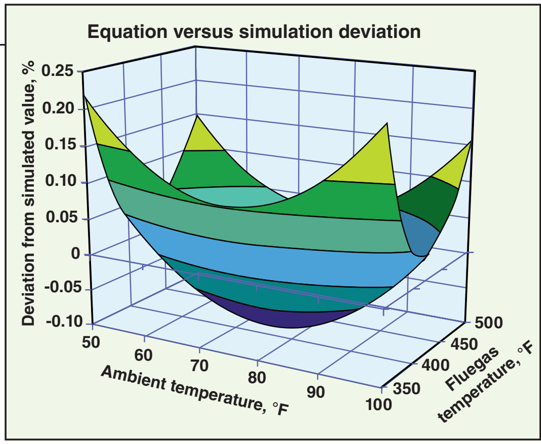


FIGURE 3. This chart compares the accuracy of the net power recovery estimated using Equation (1) versus the net power recovery as predicted by the simulation model. As shown, the results predicted by the simulation and the results predicted by the equation differ by no more than -0.1% to +0.23%

TABLE 1. REFINERY WASTE-HEAT-RECOVERY POTENTIAL

Refinery size, 1,000 bbl/d	Net power recovered, MW	Value of recovered power, $ millions/yr
500 to 600	22 to 27	up to 12.9
400 to 499	18 to 22	up to 10.7
300 to 399	13 to 18	up to 8.6
200 to 299	9 to 13	up to 6.2
100 to 199	4 to 9	up to 4.3

and fluegas composition. The case illustrated in Figure 1 is based on a typical fluegas flowrate, temperature, pressure and composition [*1*] with an ambient air-temperature of 70°F, and assumes that natural gas is the fuel used by the fluegas source.

The curve shown in Figure 2 can be used to approximate the power-recovery rate for various fluegas inlet temperatures at the assumed ambient temperature. Note that power recovery varies linearly with fluegas temperature for a given ambient temperature and fluegas stream, allowing for easy interpolation to estimate the power-recovery rate for a specific application.

Equation (1) is derived empirically from simulation data, and can be used to estimate the potential power recovery that is possible when using a simple ORC:

$$P = Q_{fg}\left[AT_{fg}T_a + BT_a + CT_{fg} + D\right] \quad (1)$$

P = Net recovered power, kW
Q_{fg} = Fluegas flow, million std. ft^3/h
T_{fg} = Fluegas inlet temperature, °F
T_a = Ambient temperature, °F
A = –0.00411 (unitless constant)
B = 0.775 (unitless constant)
C = 1.122 (unitless constant)
D = –211.63 (constant)

This equation produces estimates of fluegas temperatures that have an accuracy of ± 0.23% between 350 and 500°F with ambient temperatures varying between 50 and 100°F. Figure 3 illustrates the percent difference in the values calculated by the equation versus those produced by simulation.

Equation (1) also assumes that the cycle is running in the working fluid's subcritical region. The subcritical region is the ranges of temperature and pressure below the fluid's critical point — that is, where distinct liquid and vapor phases exist. Note that this equation is only provided for quick estimation purposes. More accurate results can be easily achieved by simulating the simple process.

Using Equation (1) to obtain empirical data on fluegas rates and temperatures for various refinery units, it is easy to formulate a table such as that shown in Table 1. Based on the size of the refinery, the potential power recovery and the associated dollar value of the power recovered can be approximated with suitable accuracy. For the case discussed here, the reported dollar values of the power recovered annually are based on a plant on-stream factor of 8,000 hours per year, and a conservative, per kW-h value of $0.06.

Various ORC configurations

Although, the main focus of this article is on the use of a simple ORC configuration, specific modifications may be made to the cycle and to improve the heat-recovery efficiency and increase the quantity of power recovered. For example, as shown in Figure 5, if an additional heat exchanger (*E-102*) were provided to preheat the refrigerant stream entering the evaporator (*E-100*) by heat exchange with the turbine exhaust stream, then an increase in power recovery of at least 10% over the simple cycle may be realized. Depending on the size of the specific installation, this could prove to be a very significant increase.

However, while improved heat-recovery efficiencies and the potential to increase the quantity of power recovered are always gratifying, the addition of extra equipment requires additional capital, operations and maintenance expenditures. These additional costs must be weighed against the value of the power recovered. Thus, depending on the required payback time and forecasted energy prices, the addition of a simple ORC system may prove to be the most cost-effective solution for a particular application.

Choice of a working fluid. The simulated properties of the working fluid used in this study are based on the physical properties of the refrigerant R-245fa (1,1,1,3,3 – pentafluoropropane), which has the trade name Gen-

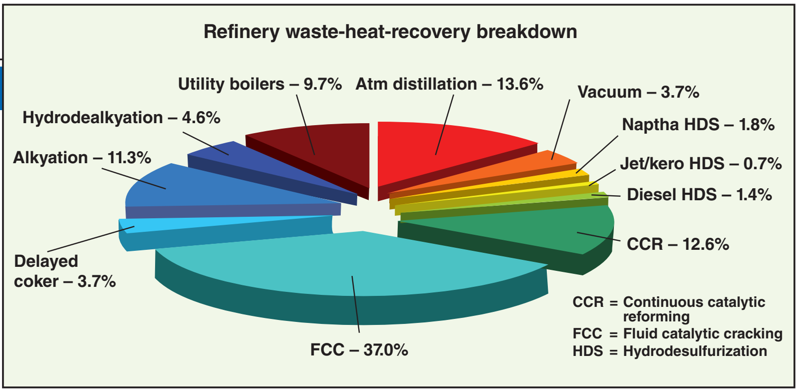


FIGURE 4. In this breakdown of waste-heat recovery by refinery unit, one can see that particularly high rates of potential waste-heat recovery are possible for fluid catalytic cracking, atmospheric distillation and catalytic reforming units and utility boilers

etron and is a product of Honeywell [*2,3,4*]. The characteristics of R-245fa make it particularly suitable for fluegas heat-recovery applications using ORC technology. This refrigerant has the required thermodynamic properties — namely, relatively high critical and condensing temperatures — which are necessary for operating in the temperature range between typical fluegas temperatures and the ambient temperature of the heat sink.

This particular refrigerant also has a relatively high decomposition temperature, which is desirable for heat exchange with hot fluegas streams. Furthermore, R-245fa has desirable environmental traits that make it a refrigerant of choice for many industrial refrigeration systems, in light of environmental concerns related to the ozone layer and fugitive emissions. For instance, the fluid has a zero ozone-depletion potential (ODP), a relatively low global-warming potential (GWP), and it is not considered a volatile organic compound (VOC) in the U.S. [*4*]

The economics and heat-recovery potential of an ORC are a function of the thermodynamic properties of the selected refrigerant. Thus, the selection of the best working fluid for a given application is important to the successful implementation of any waste-heat-recovery system. Refrigerant blends may also be considered, and the choice of refrigerant or refrigerant blend must be optimized on a case-by-case basis. Designers and marketers of ORC systems and their component machinery have process simulations and empirical data that are needed to accurately model and optimize ORC systems specific to any given fluegas stream.

It is tempting to choose a working fluid based solely on the desire to maximize the power-recovery efficiency, but there are other factors to consider, as well. For instance, depending on the specific application and location of the installation, it may be necessary to select a working fluid that is environmentally friendly, safe, and economical in addition to being inexpensive and readily available.

Air-cooled heat exchangers vs. cooling water. The results of this study are based on the assumption that in the particular application, air-cooled heat exchange is more economical than systems based on cooling water. Air coolers, depending on the cooling duty required, are generally not high energy consumers but they may require a significant amount of plot space. The decision of which type of heat exchange methodology, in terms of capital cost, operating cost, operating logistics and potential impact on the overall heat-recovery potential, must be evaluated on a case-by-case basis for each application.

Acid-gas condensation issues. When establishing the temperature at which the fluegas will exit the waste-heat-recovery unit, consideration must be given to the potential for acid condensation. This occurs when the water vapor in a fluegas stream is allowed to condense, carrying with it any compounds that may form an acid when combined with water.

In most cases, industrial fluegas streams are produced by the combustion of a fossil fuel. In many cases, this fuel contains some form of sulfur, which will likely be converted to some form of SOx during combustion. If cooled below the acid dewpoint, these combustion byproducts, along with carbon dioxide, can combine with the water vapor present in the fluegas stream and form corrosive acids [*5*].

To prevent equipment corrosion, any item that comes in contact with the cooled fluegas stream must be constructed of special materials that can withstand damage from such acids, and these special materials can add considerably to equipment costs. Such fluegas systems are also commonly designed to remain above the acid dewpoint to avoid corrosion.

While these design and operating considerations can add considerably to the overall system cost, the condensation of fluegas provides additional opportunity for energy capture, because as the water vapor in the fluegas stream condenses, the latent heat of vaporization of the water will be transferred to the working fluid, thereby increasing the potential power recovered via the ORC system.

An economic analysis must be performed to balance the tradeoffs and determine whether cooling the fluegas stream below its acid dewpoint and capturing the extra energy is worth the additional capital necessary to implement the metallurgical requirements to withstand the potential for acid attack.

Plant economics and air permit benefits of ORC technology. If properly designed, the use of an ORC system can provide a facility with an opportunity to produce additional electric power without an increase in air emissions. As a general rule of thumb, any combustion-related facility can benefit from ORC installations on furnace stacks if electrical power from external sources is limited, expensive, or unreliable by providing the plant with internal power-generation capabilities and an additional source of electricity for captive use at the site.

It is also worth noting that facilities in certain regions face restrictions on air emissions that make it costly or impossible to amend air permits to add furnaces, cogeneration units or other means of increasing utility power, a situation that can limit expansion and modernization opportunities. However, the installation of an ORC system on existing furnaces allows for the generation of electrical power without increasing air emissions by capturing waste heat from furnaces already in operation.

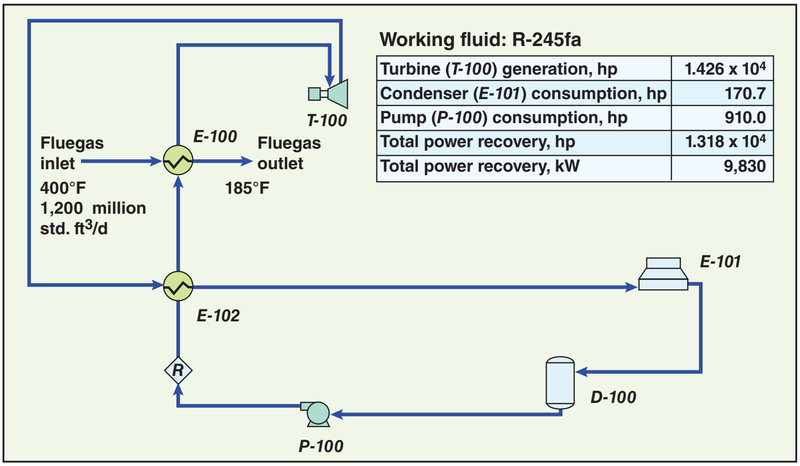


FIGURE 5. The addition of a pre-heater unit (*E-102*) to this ORC configuration helps the system enhance the overall heat-recovery efficiency and increase the quantity of recovered power

A typical ORC installation will usually generate a significant surplus of electricity compared to the power that its own equipment uses, so an ORC system's net utility costs are generally less than zero. An ORC system is therefore a minimal contributor to a plant's overall operating costs, which is another advantage of using ORC technology to improve both fluegas treatment and onsite power generation.

Final thoughts

Ongoing political developments are expected to bring about stricter enforcement of environmental regulations and increased incentives to reduce fossil-fuel consumption and imports, while the escalation of energy prices is tightening profit margins for all chemical process and refinery operators. ORC systems offer a promising means of enhancing waste-heat recovery from fluegas in both existing and new facilities, and thus help to improve the overall economics of fluegas treatment.

Today, thanks to advances in refrigerants (and blends), and ongoing innovations in heat exchange technology, metallurgy, and simulation tools, the classic ORC system can be optimized to fit ideally within a wide range of fluegas sources and treatment systems. ■

Edited by Suzanne Shelley

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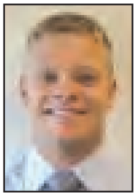

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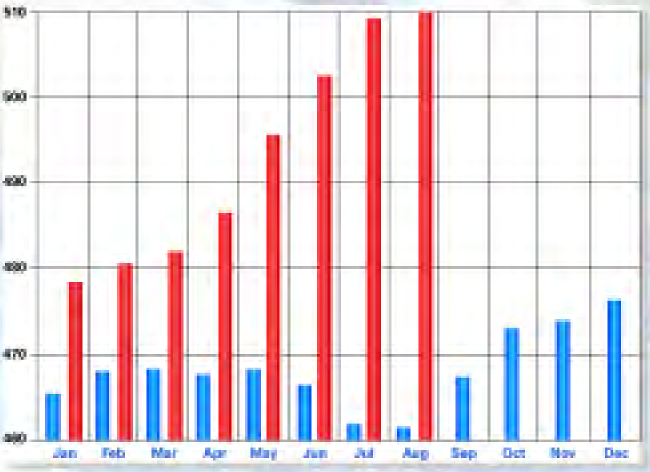

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Selecting Laboratory Exhaust Systems

With upfront thought, exhaust systems can ensure personnel safety and improve building efficiency

Paul A. Tetley
Met-Pro Corp.

FIGURE 1. Roofline aesthetics are among a host of considerations when selecting a fume-hood exhaust system for a laboratory facility

As components of chemical research facilities, fume-hood exhaust systems for laboratory workstations are often taken for granted. Yet the choice of exhaust system, when designing or specifying a laboratory workstation, can have a tremendous impact on the safety of the laboratory building's occupants as well as those in adjacent buildings. The choice of a fume-hood exhaust system can also affect a building's longterm operating costs.

A better upfront understanding of the various types of exhaust systems and their operation can eliminate potentially significant safety problems in the future while optimizing the efficiency of the building and the reliability of the system.

Exhaust system basics

The main purpose of a fume-hood exhaust system in a laboratory is to remove contaminated air resulting from chemical fumes, reactions and other laboratory activities. The system must provide maximum protection while using the least possible amount of air. It must control the flow of two types of air: workstation exhaust, and the clean air that replaces it.

When designing new laboratory facilities, or upgrading existing ones, safety — with regard to re-entrainment of workstation exhaust into the building or adjacent buildings — and indoor air quality to protect building occupants, are the most important considerations for a fume-hood exhaust system. In addition, the system's ability to comply with appropriate pollution-control standards (including elimination of unpleasant odors in the discharge stream) is also a key consideration.

Other important considerations include: system efficiency, initial and operating costs and roofline aesthetics (Figure 1). For example, quiet, vibration-free performance can be important to protect delicate laboratory instruments.

Before selecting a laboratory workstation fume-hood exhaust system, the location of the workstation should be carefully thought out. Its location can significantly affect efficiency and safety. For example, occupants in the laboratory can create air turbulence simply by walking past the workstation, causing contaminants to be pulled outside of its air space. For a workspace designed with an air diffuser directly above the fume hood, significant air turbulence can cause contaminants to escape into the laboratory.

In addition, airflow into the laboratory will affect the performance of a fume-hood exhaust system. Since the laboratory should maintain negative air pressure with respect to the outside, the doors, windows, air vents and other openings should remain closed during normal operation to ensure that any contaminants will be exhausted through the fume hood, rather than by other laboratory openings.

The effectiveness of a laboratory fume-hood exhaust system can be gauged in terms of safety provided. The principle design parameter is to allow safe removal of fumes away from the operator and from the occupants of the laboratory. The velocity of the air entering the hood from the room is the key factor in achieving this safety. If the velocity is too low, the fumes can escape the hood and enter the room. If the velocity is too high, air turbulence at the sash (damper) can occur, which may push fumes into the room. Airflow velocities that optimize safety can vary by manufacturer, but typically are in the range of 100 ft/min.

Exhaust flow from a fume hood should be greater than the supply air to create outside-in air movement and thereby contain airborne contaminants.

Workstation fume-hood exhaust systems can be either constant-air-volume (CAV) or variable-air-volume (VAV) designs. A CAV fume hood exhausts air at a constant rate, with an airflow velocity that increases as the sash is lowered. A VAV laboratory fume hood attempts to eliminate the variable velocity by varying the air volume exhausted through the hood. In doing so, it can reduce the total supply of exhaust air, and contribute to reduced operating costs. VAV fume hoods use a closed-loop exhaust system to monitor the amount of air being exhausted in order to maintain the required average face velocity. The control system also helps monitor the presence of contaminants. In addition, many VAV hoods use visual and audible alarms and gages to warn of inadequate air velocity or exhaust system malfunctions.

Exhaust system fans

Laboratories can choose from a number of fume-hood exhaust-system designs based on the requirements of the facility. Exhaust systems rely on hidden, but essential subsystems, including exhaust fans and ventilation systems.

Fume-hood exhaust systems typically employ a network of air ducts (plenums) leading to a roof-mounted exhaust system that is driven by one of three fan technologies: axial, cen-

FIGURE 2. A difference between exhaust system types is the height of rooftop stacks, a characteristic with value in accommodating space and weight restrictions

trifugal, and mixed-flow impeller. The fans in each of these categories can be direct-drive designs, with the fan wheel mounted directly on the motor shaft, or belt-driven, with the fan wheel mounted on a separate drive shaft and driven by power from the motor transferred through one or more belts.

Axial fan systems Axial fan blades draw air into fan housings and discharge air in the same axial direction — ideal for moving large volumes of air, rated at cubic feet per minute (ft^3/min) at low pressures, and also rated in static pressure (SP). Fan blades used in axial fans are typically plastic, aluminum, stainless steel or fiberglass. Different types of axial fans used for industrial applications include panel fans, which are wall-mounted to draw air into — or exhaust air out of — a room, and tube axial fans, which are connected to a series of ductwork conduits for exhausting contaminated or hot air from a location.

Centrifugal fan systems Centrifugal fans have blade wheels mounted inside a housing. The blades can be forward-curved, backward-curved or straight. Exhaust air is pulled into the inlet of the blower housing and deflected by the fan blades into a spiral pattern. This pattern of airflow is then focused by the scroll housing and directed into a single outlet airstream. The airstream leaves the fan's housing at a right angle to the axis of the housing assembly. A variation on the traditional, centrifugal scroll-type fan housing is the tubular centrifugal fan, which uses a cylindrical housing rather than a scroll housing and cutoff assembly.

A centrifugal fan is designed to work at fairly high revolutions per minute and with a tall, roof-mounted exhaust stack in order to discharge the exhaust air at sufficient height above a facility to prevent re-entrainment of exhaust air (and its possible noxious fumes or unpleasant odors) into the building and surrounding facilities. Because of the size and height of the stack, it requires expensive and complex mounting hardware for stability and to minimize vibration. This includes elbows, flex connectors, spring vibration isolators and supporting guy wires.

Mixed-flow impeller systems Mixed-flow impeller systems generally provide greater airflow than centrifugal fans, and greater air pressure than axial fans. These systems eliminate re-entrainment of exhaust air and odors into a facility and surrounding buildings with improved indoor air quality (IAQ). This technology typically incorporates low-profile, roof-mounted exhaust systems with lower-speed fans as compared to the other technologies. To minimize noise levels, these systems employ low-vibration, direct-drive motors to draw in the exhaust air from a laboratory fume hood.

A noticeable difference between exhaust systems with centrifugal fans and those with mixed-flow impellers is the height of the exhaust stacks: mixed-flow impeller systems present a lower profile and smaller footprint on the roof (Figure 2). This characteristic has obvious value in accommodating space and weight restrictions, as well as in its aesthetic qualities (more and more communities are passing ordinances limiting overall building height, including rooftop appurtenances).

As the name implies, a mixed-flow impeller system combines the best attributes of both axial and centrifugal air movement technologies. Impellers in this approach have high efficiency, consume less energy and are typically quieter. In addition, true mixed-flow technology is stable throughout the impeller performance curve. Therefore, these impellers do not have a stall or unstable performance region, as is found on axial and centrifugal fan curves in regions of higher pressure. The concern is that systems operating at or near this unstable performance region can give rise to significant performance, sound and vibration problems.

Mixed-flow impellers blend the exhaust air from a laboratory fume hood with outside, ambient air to reduce the concentration of contaminated particulates, as measured in parts per million (ppm) or milligrams per cubic meter (mg/m^3) to a level that can be safely and legally exhausted to the atmosphere. These fans draw exhaust air into a network of ducts that transport it to the outside of the facility, usually to the roof, where it is mixed with ambient air. In some cases, particularly where local ordinances dictate rooftop appearances, mixed-flow impeller systems may be mounted in an enclosed facility (this is usually done for aesthetic reasons).

Using base-mounted bypass dampers, a mixed-flow impeller can draw in twice as much fresh air as exhaust air, resulting in a much lower concentration of contaminants and odor-causing elements to be treated from the laboratory fume hood.

Release to atmosphere

Because a mixed-flow-impeller exhaust system can generate relatively high escape velocities, even at fairly low fan speeds, additional fresh air is entrained into the exhaust mixture when it is expelled from the rooftop stack by means of a nozzle/windband section. This mechanism creates a protective envelope around the discharged exhaust-air mixture and leaves the surrounding air stationary to improve entrainment. The exit velocities of these escape gases can exceed 6,300 ft/min and can send an exhaust plume more than 120 ft into the air, even into steady crosswinds. Of course, a variety of factors can determine the effectiveness of the system, including building architecture, surrounding terrain and even weather conditions.

As an example, a mixed-flow impeller moving an exhaust and clean air mixture at 80,000 ft^3/min will typically generate 6,300 ft/min exit velocity. According to wind tunnel research, additional air is entrained into an air plume through aspiration, even at exit velocities of 3,000 ft/min (the minimum velocity specified by the ANSI Z9.5 standard for clearing chemical contaminants in laboratory ventilation systems).

A belt-driven, centrifugal-fan exhaust system and duct network can also mix contaminated and clean air similar to a mixed-flow impeller, with useful dilution effects when released into the atmosphere. But the centrifugal fan approach requires a rooftop stack with sufficient height, sometimes as high as 40 ft or more. For the same dilution and operating efficiency, a mixed-flow impeller system will typically rise only about 15 ft above the roofline. Because of the lower height, the mixed-flow impeller exhaust system would not require the extra stabilizing hardware needed with centrifugal fan exhaust systems.

Tall exhaust stacks are not the only structures associated with centrifugal fan systems. Often, centrifugal fan systems also need a rooftop penthouse, mainly to protect service personnel. Because centrifugal fan systems rely on belt-drive motors — in contrast to the more reliable direct-drive motors of mixed-flow impellers — periodic maintenance visits are required to adjust belt tension or to replace pulleys or failed belts. The penthouse enclosure maintains safe, workable conditions for maintenance personnel.

In terms of reliability, laboratory workstation fume-hood exhaust systems based on mixed-flow impellers are relatively simple and maintenance-free compared to the belt-driven fans and blowers of typical centrifugal and axial fan systems. The belts alone in a belt-drive system, which can stretch, loosen and break over time, require regular maintenance and repair, resulting in shorter expected operating lifetimes. For example, the minimum expected lifetimes (L_{10}) of bearings in belt-driven systems are typically 40,000 h, while the expected operating lifetime of direct-drive motor bearings can be at least 200,000 h for some mixed-flow impeller systems.

A quieter solution

Noise management is also an important consideration for most laboratories and areas — in some cases requiring firms to perform noise studies at their property lines to determine the noise levels of a facility's various systems, including the laboratory exhaust system. The lower speeds and inherently quieter design configuration of the direct-drive motors in mixed-flow-impeller exhaust systems, compared to axial and centrifugal fan systems, usually translate into lower noise and vibration levels. In addition, for applications where noise must be tightly controlled, mixed-flow impeller systems can be designed with integrated noise attenuators that do not increase the overall height of the roof exhaust fan.

Axial and centrifugal fan exhaust systems can also be equipped with noise-reduction attachments, such as a discharge silencer, added onto the exhaust stack. But this adds to the already tall stack height, and requires additional mounting hardware to minimize vibration induced in the building. Even at their lower motor speeds, there is little sacrifice in performance with a mixed-flow impeller. It can provide greater airflow than a centrifugal fan, and greater air pressure than an axial fan, at lower rotational speeds.

Compared to belt-driven centrifugal and axial fans, direct-drive mixed-flow impeller fans are energy-efficient and cost-effective choices for fume hoods, with about 25% greater energy efficiency than belt-driven centrifugal fan systems. Improperly aligned shafts, pulleys, pillow blocks and motors in a belt-driven system can rob it of power, and can introduce reliability issues. Also, the outlet nozzle of a mixed-flow impeller system can be designed for high exhaust-gas exit velocities even at lower fan speeds and corresponding reduced power requirements.

For further energy efficiency, it may be necessary to specify a variable-speed drive system. The use of variable-speed drives can contribute to lower energy costs, running at slower fan speeds at night when laboratories are unoccupied. In addition, if one fan fails in a multiple-fan exhaust system, the remaining fans increase speed to compensate. This is critical in many biosafety level (BSL) laboratories, where redundancy is mandatory.

In addition, energy savings are also possible by recovering some of the heat normally lost through exhausted air. Mixed-flow impellers can be equipped with unique heat-recovery modules that extract warmed air from the exhaust flow and transfer the heat energy to the intake of a building's ventilation system, significantly reducing heating fuel costs for the building (for every 1ºF in added heat, there is a corresponding 3% reduction in heating fuel costs).

Ductless laboratory fume hoods

The three major laboratory fume-hood exhaust systems discussed so far assume the use of ductwork networks to vent contaminated air from the laboratory. In recent years, ductless laboratory fume hoods have emerged as lower-profile alternatives. In ductless designs, the hood incorporates a carbon-bed or other type of filter to remove and control contaminants. The filter media must be matched to the types of chemicals used in a laboratory. General-purpose filters will adsorb a percentage of most chemicals and gases, while particulate matter filters may be needed for some applications. In some cases, a chemical impregnate may be needed in the filter to alter the target chemical to a less hazardous material.

A ductless, laboratory fume hood has no external connections other than its power cord, making it extremely portable and useful for facilities that require more than occasional redesign. The power cord connects to electronic systems that monitor airflow and filter conditions for operator safety, as well as provide visual and audible alarms when needed. A ductless approach is not a substitute for all ducted systems, and should never be used in any chemical laboratories involving unknown results (for which the filter may not work). Admittedly, ductless laboratory fume hoods sacrifice efficiency for portability, especially compared to a centrifugal or mixed-flow impeller system, and they require careful monitoring of the carbon filter's condition so that replacement can occur once the filter has become saturated. ■

Edited by Scott Jenkins

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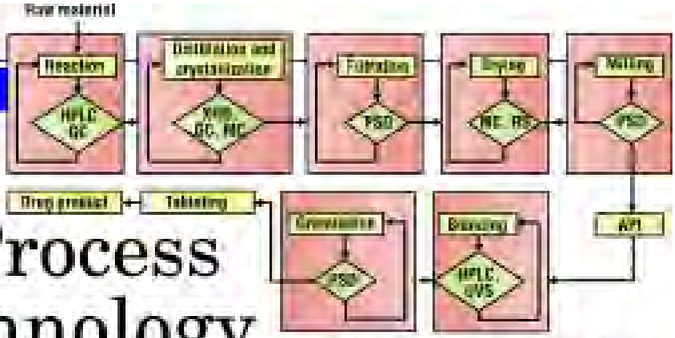

FIGURE 1. In conventional pharmaceutical production, quality analysis typically follows manufacture of the finished product and is done offline

Counting On Process Analytical Technology

Affordable healthcare will require drugmakers to make better drugs more efficiently

Sanjay Gade and **Deepak Jain**,
Ranbaxy Laboratories Ltd.

If the debate that culminated in passage of national healthcare reform in the U.S. is any indication of what is ahead for pharmaceutical manufacturers, they should ready themselves for battle. Implementation of the sweeping measure puts the onus on drugmakers to minimize waste and boost production of active pharmaceutical ingredients (APIs) and finished products, ensuring affordable healthcare for everyone.

Since 2002, when the U.S. Food and Drug Admin. (FDA) introduced the Process Analytical Technology (PAT) program, the agency has pinned high hopes on the prospect that the voluntary initiative would drive innovation in pharmaceutical design, development and production [1]. While drugmakers' response to PAT has been positive, it has not translated into significant action. Skeptical that drugmakers will ever be so motivated, some policy makers are pressing FDA to make PAT mandatory.

A batch of tradition

Conventional pharmaceutical manufacturing is generally accomplished using batch processing with laboratory testing conducted on collected samples to evaluate quality (Figure 1). While this approach has been successful in producing quality pharmaceuticals, it has several drawbacks:

- Repeated process optimization requirements
- Recurring manufacturing difficulties
- Unexpected batch failures, hence higher waste generation and manufacturing costs
- Uncertain product quality

However, significant opportunities exist for improving pharmaceutical development, manufacturing and quality assurance through innovations in product and process development, process analysis and process control. Gains in quality, safety and efficiency are likely to come as processors advance efforts to:

- Reduce production cycle times by using on-, in- or at-line measurements and control, or a combination thereof
- Prevent rejects, scrap, and reprocessing
- Employ realtime release as test method for ensuring final product quality
- Increase automation to improve operator safety and reduce human error
- Facilitate continuous processing to improve efficiency and manage variability
- Use small-scale equipment and dedicated manufacturing facilities
- Improve energy and material use and increase capacity

PAT involves the application of continuous monitoring and control of manufacturing processes so that parameters adjust themselves to reach the desired optimum state [2]. It involves a fundamental shift from testing the quality of raw materials to building quality into products through realtime — online, inline, and noninvasive — analysis (Figure 2). Table 1 lists the primary tools and their applications to specific unit operations.

Raw material analysis

Pharmaceutical manufacturing involves an array of raw materials (average 30–35 per process), including reagents, solvents, catalysts and auxiliary chemicals in different phases of solids, liquids and gases.

While the quality of raw material varies with batch number source, age, packaging, storage and handling, there is a some slight variation in the data due to differences in the analytical instruments or sampling techniques. The make or model instrument used for gas chromatography (GC) or (high pressure liquid chromatography (HPLC) can account for fluctuations in run times, flowrates, pressures or column responses. Depending on the material characteristics and sampling hardware facilities, direct or indirect sampling techniques can be specified. If the material is sensitive to humidity, oxygen or light, sampling errors due to exposure can cause analytical deviations.

Quantifiable, causal and predictive relationships among the raw materials, the manufacturing process, and final product quality are essential for the development of robust and rugged processes. PAT tools, such as near-infrared (NIR) spectroscopy, make such processes possible.

Reaction monitoring

Standard analytical methods for reaction monitoring employ chromatographic resolution to separate mixtures. Such methods have a high degree of accuracy, but the longer the period between sampling and analysis, the greater the potential for error. Analysis is typically conducted offline, with run times of 30 min to 2 h after the sample has been removed. Samples are sometimes stored and analyzed, and in some cases, analysis time is longer than reaction time, especially for fast reactions of about 1 s to 30 min, which may result in overreaction and formation of unwanted side products. The resulting batch failure requires repetition of reaction, which leads to reduced efficiency and quality.

A PAT alternative for analyzing a liquid reaction mixture is to use NIR and an inline fiber-optically coupled

TABLE 1. KEY PAT TOOLS AND APPLICATIONS	
PAT Tool	Application in Unit Operations
Near Infrared Spectroscopy (NIRS)	Raw material testing, Drying, Powder blending, Coating
Fourier Transform Near Infrared Spectroscopy (FT -NIR)	Mixing, Reaction, Solvent recovery
Attenuated Total Reflectance (ATR), Particle Video Microscopy (PVM), X-ray diffraction (XRD), Raman Spectroscopy (RS)	Crystallization, Drying, Powder blending, Coating
Heat Balance Calorimetry	Reaction, Crystallization
Laser Diffraction (LD)	Crystallization, Milling, Granulation
Digital Image Processing(DIP)	Crystallization, Milling, Granulation
Dynamic Light Scattering (DLS)	Crystallization, Milling, Granulation
Tomography	Mixing, Filtration, Drying
Turbidity Metering	Crystallization, Filtration
Conductivity Metering	Filtration
Thermodynamic Lyophilization Control (TLC)	Lyophilization
Acoustic-Resonance Spectrometry (ARS)	Tableting

flow cell or transmission probe that requires neither sample preparation nor laboratory analysis [3]. The process can be more tightly controlled, problems detected earlier, and overall product quality and yields improved.

Many chemical reactions used in the manufacture of APIs use metal catalysts. In some cases, these metal catalysts are doped with carbon black particles to increase the catalyst surface area. The high sensitivity and throughput of a Fourier-transform near-infrared (FT-NIR) system can analyze these solutions with carbon-black concentration levels of 0.05–0.5%. Catalytic hydrogenation reactions are potentially sensitive to matrix effects. Inline analysis provides kinetic and mechanistic information that can be used to study the sensitivity of the reaction to matrix effects.

Optimal mixing

An agitator impeller can be designed and operated such that agitator power input is used to maximize flow with minimum shear, maximize shear with minimal flow, or to balance shear and flow at a designated point in between. However, solid-solid, liquid-liquid or solid-liquid mixing or combinations thereof are complex. For these applications, high-shear mixers are typically used because they can combine immiscible fluids to produce emulsions and dispersions through the controlled formation and integration of droplets. They are also used for deagglomeration and particle size reduction to improve the blending of solid ingredients into liquids.

Many powders are difficult to disperse efficiently and the resulting mixtures are prone to persistent clogging, which tends to drive up costs and drive down throughput. Early indication of these problems can control or eliminate them.

Tomography involves taking measurements around the periphery of an object, such as a process vessel or impeller, to determine what is going on inside [4]. Each image (tomogram) is composed of an array of derived conductivity measurements. Process vessels with all types of mixers and accessories can be retrofitted with a linear tomography probe, typically a baffle, to measure axial mixing and solids concentration levels. The drug and the diluents can be dry-mixed in a double cone mixer, which gives ease of cleaning and complete discharge. The homogeneity of the mixture can be confirmed using NIR measurements with an FT-NIR spectrometer.

Solvent recovery

The solvent recovery process involves the distillation of a feed or crude solvent from a low purity of about 60%, to a pure product that can be reused in the manufacturing process. This process is commonly analyzed by offline GC, requiring an operator to collect a sample from the process and take it to a laboratory for analysis. This process can take longer than 1 h and result in the distillation of a product that does not meet specifications.

Process FT-NIR has many benefits over GC in this application, and, if implemented correctly, can give realtime analysis of the feed streams and the product [5]. High-quality NIR spectra can be collected usually in less than 1 min and the quantitative analysis of multiple components performed. This information is then used as a key component of the control strategy to change column temperatures or reflux ratios in either a feedback or feed forward control. This results in better product quality and yield.

Crystallization

Pharmaceutical crystallization processes are extremely complex and are not baseline scalable. The key aspects of a crystallization process are the rate of nucleation and growth rate of the crystals. Both of these rates depend on the type of flow, mixing, heat and mass transfer rates of the process in an unpredictable manner. Impurities and additives can have a significant effect on these rates by altering the interfacial energy of particular crystal faces. The main measures of the outcome of a crystallization process, listed here, are difficult to reproduce and achieve.

- Particle size distribution, or the relative amounts of particles present, sorted according to size
- Crystal shape (needle, cube and plates)
- Polymorphic form — polymorphism is the tendency of some compounds to exist in more than one stable crystal form

The size and shape of the crystals formed has a strong effect on processability. Smaller-sized and nonspherical particles make flow, filtration and processing more difficult. Fine particles have greater surface area and can collect more impurities at the surface. The solubility of different polymorphic forms may differ, and dissolution rates will depend on crystal size as well as polymorphic form.

Supersaturation measurement is the key process variable that affects crystallization performance [6]. To control supersaturation, the solubility curve and meta-stable zone width (MSZW) need to be known.

Focused-beam reflectance measurement (FBRM) can be used to measure the solubility curve and MSZW by identifying the point of dissolution and point of nucleation at different solute concentrations. FBRM provides information about yield, suitable seeding locations and optimal zones of operation to get uniform particle size. MSZW depends on cooling rate, agitation and solid concentration.

Uneven or different particle-size distributions may require further powder processing, such as milling or compaction. In some cases, the additional processing costs can be reduced or eliminated through realtime observation of crystal size distribution using laser diffraction (LD), and the progress of crys-

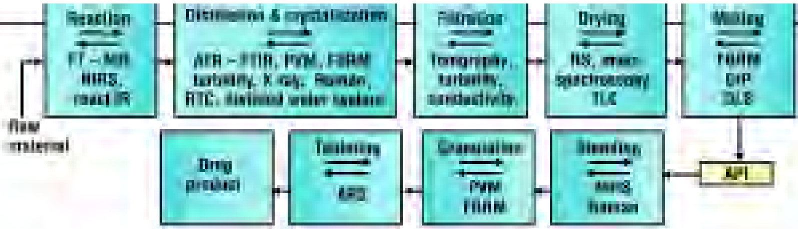

FIGURE 2. In this PAT-enabled continuous process, production is monitored and controlled in realtime, so that parameters adjust themselves for optimum results

tal shape formation can be monitored using digital image processing (DIP) and dynamic light scattering (DLS).

Faster filtration

Variations in particle size can change cake resistance and thus, filtration time. When batches are processed in series, poor process control can vary batch times, and, in some cases, lead to batch failure. Filtration, done in a batch or continuous mode, is controlled by adjusting the filter media and managing cake thickness. Analysis for particle size and concentration of particles in the feed fluid-solid mixture is offline, making process monitoring difficult [7].

Tomography has been used in a variety of separation processes to identify the changing concentration of two phases as a function of space and time. In the case of pressure filtration, a tomographic sensor can be used to observe the drying cycle with the objective of reducing time and improving the quality of the process.

Online turbidity measurements provide data on the concentration of undissolved, suspended particles present in a liquid phase and online conductivity of total ionic concentrations in aqueous solutions. The determined particle concentration is used for process monitoring and optimization.

Drying

Drying influences the solid state characteristics of APIs. Minor changes in operating conditions (temperature, pressure, vacuum and humidity), raw materials and additives, and the type of dryer and its setting can wreak havoc in many ways:

- Changes in the polymorphic form of the active ingredient
- Formation of degradation products (thermal, oxidative, moisture and light)
- Variable solid properties (particle size distribution, bulk density, compressibility, angles of repose and friction; generation and agglomeration of fines, and loss on drying (LOD) variations
- Static charge development
- Thermal instability leading to hazardous runaway situations

Generally the standard operating method involves either stopping the process and withdrawing a sample for primary analysis, or drying the product for a fixed period of time. Because these methods do not provide continuous monitoring of the drying process, an uncertainty of consistency and scalability exists.

It is prudent to develop PAT-based drying technologies that can provide realtime control of the parameters affecting solid state properties. NIR spectroscopy in combination with an inline, diffuse reflectance measurement of the filtered material provides a means of continuously monitoring the drying process. Also, online Raman spectroscopy can continuously monitor API solid-state form during drying. Mass spectrometry facilitates inline sampling with a portable probe and helps make endpoint determinations, such as LOD or moisture content (MC).

Thermodynamic lyophilization control (TLC) enables PAT compliance in lyophilization. It determines the end of primary and secondary drying, and controls the entire process by adjusting operating parameters.

Shape up for milling

A less than optimal comminution or milling setup [7] causes a multitude of operational problems in downstream pharmaceutical processes because the particle size and shape influence a large variety of important physical properties, manufacturing processability and quality attributes, including the following:

- An excess of fine particles increases the respiratory hazard and risk of explosion. Large particles tend to flow well and can be relatively easy to control
- Dissolution rate and bioavailability of APIs
- *In vivo* particle distribution and deposition, absorption rate and clearance time, especially for aerosols and colloid systems designed for targeted drug delivery
- Content and dose uniformity and other properties related to physicochemical stability
- Aerosolization for the dispersal and delivery of a drug as an aerosol
- Flow and packing properties, powder mixing and segregation, rheological characteristics of liquid and semisolid formulations
- Grittiness of solid particles in chewable tablets and dermal creams, and irritability of ophthalmic preparations

A lack of realtime particle-size monitoring during comminution leads to significant process losses, too. There are number of positions in which online particle-size analyzers, based on technologies such as LD, DLS, DIP, and focused beam reflectance measurement (FBRM), can be used for closer control of the milling process.

Blending powders

The blending of API with excipients is a critical step in the manufacture of pharmaceutical solid-dosage forms [2]. Without a homogenous blend with the accurate and exact API content, it is impossible to get uniform doses.

Blending occurs with three independent mechanisms: convection, dispersion and shear. Low shear, symmetry in design and systematic loading are essential components of the blender. Many granular mixtures can spontaneously segregate into regions of unlike composition when perturbed by flow, vibration and shear. Once a good blend is achieved, the mixture must still be handled carefully to avoid any segregation that might occur.

Typically, samples are removed from the blender bin by so-called thief, and then analyzed by HPLC or UV-Vis spectroscopy. Care and skepticism have to be employed when relying on thief probe data. Apart from the fact that only the distribution of API is determined by assumption of the excipient's homogenous distribution, there is a significant risk of sampling error. Thief probes have been demonstrated to induce large sampling errors due to poor flow into the thief cavity, and sample contamination from the other zones of the blender during thief insertion.

In classical offline chemical methods,

dispersion can be achieved in a liquid cell with the addition of appropriate surface-active agents. Dispersion can also be achieved by controlled agitation or by the application of ultrasound. Here, there is always a possibility of changing the particle-size distribution due to the thermodynamic Ostwald ripening or recrystallization effects. In addition, the blender is stopped at fixed intervals for sampling. This process of interruption of the blend cycle and repeated sampling may change the state of blend.

NIR spectroscopy is a fast and nondestructive method to determine blend homogeneity of all the compounds and endpoints in blending. Online Raman spectroscopy can evaluate the effect of parameters such as blend time, blender speed, API placement in the blender, filler particle size and density, multiple tablet components, and sample homogeneity. The tool is feasible due to large number of samples across the run (sampling every 20 s) and the availability of spectral information on all blend components throughout the process.

Controlled granulation

The purpose of granulation is to convert light, small powders into high-density free-flowing granulates. A number of phase changes or process-induced transformations can occur due to the different process steps of wet granulation, which include wetting, mechanical stress and drying.

Granulation or the manufacture of pellets using the extrusion-spheronization technique includes several process stages (blending of the dry mass, wet granulation of the mass, extrusion of the moist mass, rotation of the extrudate by spheronization, and drying). Consequently, depending on the drug substance and excipients processed, solution-mediated polymorphic transformations probably take place. A lack of particle size measurement in granulator processes denies cost savings and product consistency.

In granulation, particle size is critically dependent on the amount of liquid. Using inline particle-size analyzers, variables such as moisture content and liquid saturation of the agglomerate can be controlled for desired particle size [7]. An image-processing system with particle vision management (PVM) can be used as a PAT tool to accurately control granule growth in high-shear granulation processes.

Tableting

Conventional tableting presses are multistage cycling processes that employ punches and dies. These machines are more expensive to operate and less efficient to use than systems based on acoustic-resonance spectrometry (ARS), which measures sample compaction, axial strain, deformation, hydration, drying endpoint, elasticity, molecular stacking, and homogeneity [*8*]. ARS identifies and quantifies an analyte online, providing a simple and continuous method of process control for tableting operations.

Perfect coatings

To be coated, tablets are loaded in large rotating pans and vented for hot air drying with coating-material-filled intagliations. Intagliations are impressions typically achieved by engraving or impressing an identifying mark or logo onto a tablet or other solid dosage form. Because edges and corners cannot be coated to the same thickness as the tablet face, the final product may appear to be nonhomogenous, imperfect and not finished at all.

NIR spectroscopy is used as an at-line technique for monitoring tablet film coating in a Wuster column. The is also useful for analysis of the active layer of film-coated tablets, for inline analysis of film coating of pharmaceuticals pellets in a fluidized-bed apparatus and for inline monitoring of tablet film coating in a pan coater.

Raman spectrometry is used as a tool to examine the variability of tablet coatings by a spectral changes correlation with coating thickness [*9*]. Here, a probe operates with a revolving laser focus to average content and coating nonuniformity.

PAT potential

PAT usage encourages the application of process engineering in pharmaceutical manufacturing and regulatory assessment. PAT, therefore, holds the great promise of improving productivity, efficiency and quality of pharmaceutical intermediates, active ingredients and finished drug products. ■

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Connect lines quickly with this coupling system

The new CN Series of multiple clean-break quick-coupling systems (photo) is designed for industrial filling systems where a mix-up of media could have dire consequences. The coupler features a cam-lock action, which facilitates the connection, even at residual pressure. The operator only has to turn the coupling by 120 deg to lock it and to quickly connect the lines. During this rotary motion, the valves are opened. Very little effort is needed for connection since the axial forces required to operate the internal valves are reduced by the cam action. The CN Series is suitable for the transfer of aggressive or hazardous fluids. These couplings are available in nominal sizes of 25, 40 50, 80 and 100 mm. Depending on the size, the nominal pressure ranges from 10 to 25 bar. — *Walther-Präzision, Carl Kurt Walther GmbH & Co. KG, Haan, Germany*

www.walther-praezision.de

Bulk bag fillers with even more throughput than before

Next month at Pack Expo 2010 (Chicago, Ill., October 31–November 3), this firm will exhibit one of its latest Bulk Bag Fillers (photo) with new features that increase the number of bags that can be filled per hour while reducing operator fatigue and maintaining filling accuracy — even for NTEP resale applications. Many standard operating features are included in all the firm's weigh fillers, enabling the processor to begin immediate production without the need for extra equipment. In addition to the built-in features of the standard units, a full range of options is provided, allowing many other operations to be performed from the same machine. — *Spiroflow Systems, Inc., Charlotte, N.C.*

www.spiroflowsystems.com

This scissors lift has a small footprint

The DXL Series Compact Scissors Lift (photo) features a smaller footprint than standard lifts, while offering a full 2,000 lb of lifting capacity. The double-scissor mechanism allows for a low 6-in. collapsed height while providing a 42-in. raised height. A foot switch operates a 1/3 h.p. motor. The unit comes with an 18 in. x 30 in. platform as standard. — *Presto Lifts Inc., Attleboro, Mass.*

www.prestolifts.com

Note: For more information, circle the 3-digit number on p. 62, or use the website designation.

Flexicon

A safe way to store IBCs

This firm has introduced a new product line engineered specifically for the storage of intermediate bulk containers (IBCs). With increased point-of-use, the firm designed single- and double-tote storage lockers. The units are offered in both non-fire rated and 2-h fire-rated models. Both models offer oversized sumps to accommodate the extra storage needed by the tote containers, but have also been designed specifically to keep them within the size restrictions necessary to ship via enclosed trailers. — *Safety Storage, Inc., Charleston, Ill.*
www.safetystorage.com

Container size and shape not important for this system

The Monobloc Fill/Finish Packaging System accommodates different container sizes and shapes. These systems can perform a wide range of tasks, such as sorting, feeding, filling, plugging, stoppering, crimping, induction sealing, labeling and accumulating. All Monoblocs are digitally controlled with menu-driven programs that electronically adjust operating parameters. — *Filamatic, a div. of National Instruments LLC, Baltimore, Md.*
www.filamatic.com

This dump-weigh batch system is sanitary

A combination bulk bag and manual dump batching system (photo) weighs bulk materials that are discharged from bulk bags or manually dumped from sacks, boxes and other containers, and empties the batch into mobile storage bins. The sanitary system consists of a bulk bag unloading frame, two hoppers and two flexible screw conveyors that feed a center gain-in-weight hopper under automated control. The unloader features a Spout-Lock clamp ring that forms a high-integrity seal between the clean side of the bag and the clean side of the equipment. Material contact surfaces are of stainless steel finished to sanitary standards, with the exception of removable, polymer conveyor tubes. — *Flexicon Corp., Bethlehem, Pa.*
www.flexicon.com

Improved filling-opening feature saves time, reduces film usage

This firm has teamed up with Pactiv Corp. to supply Hefty Slide-Rite slider customers with an improved filling and opening feature for heavier (15–65 lb) bagged products. The challenge for slider bags that are bottom-filled is to minimize the fill force against the slider assembly, while making it easy for users to open and access contents. Until now, packagers had been lining the entire bag with peel-seal film. The co-developed alternative uses a 1/2-in. wide strip that replaces several feet of expensive film. — *Rollprint Packaging Products, Addison, Ill.*
www.rollprint.com

A drum lifter with onboard hydraulics

This device is ideal for transporting and lifting drums, regardless of their material (plastic, fiber and mild steel). The operator has a clear view of the drum, unobstructed by a mast, making it easy to feed a production or filling line. All movements are actuated by the onboard hydraulics: picking up, lifting, lowering and inverting. Various types are available, with load capacities from 150 to 350 kg. Available on request is a motorized chassis, which provides additional mobility. — *Müller GmbH, Rheinfelden, Germany*
www.mueller-gmbh.com

Fully automatic bag loading, without pallets

With the autopac 3000, loading and palletizing system, bags can be loaded onto lorries automatically. The machine simultaneously loads and palletizes bagged goods such as cement, limestone or gypsum — without needing pallets. In rows or stacking patterns with a pre-selected number of layers, the autopac 3000 works quickly and effectively, achieving a performance of up to 3,000 bags/h. The machine can load bags in double patterns of five, six and ten bags. The autopac 3000 does not operate on a pneumatic and hydraulic drive unit, because this can lead to leakages and therefore to soiling of the load. Also, primarily in warmer countries, these energy-intense drive units require additional cooling. Therefore, for a high level of availability and efficient operation, electromechanical components are used. — *Beumer Maschinenfabrik GmbH & Co. KG, Beckum, Germany*
www.beumer.com

Mobile filling directly at the container

Either used for the filling of uncritical liquid or pasty products or for hazardous area application in Zone 1, a mobile filling station can help save costs. Complex piping and hosing can be spared as these mobile "Plug & Fill" machines are directly brought to the tank — either by rollers or via floor conveyor. No foundation is necessary. The same machine can be used at many different locations to fill different products into drums, pails, cans or IBCs. — *Feige GmbH, Oldeslne, Germany*
www.feige.com ■

Gerald Ondrey

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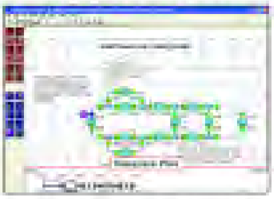

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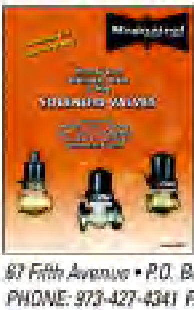

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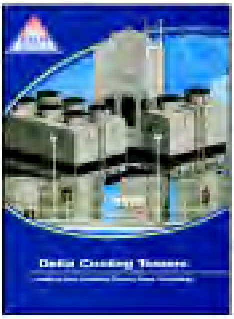
Delta Cooling Towers, Inc.



Delta Cooling Towers manufactures a complete line of corrosion-proof engineered plastic cooling towers. The towers incorporate a high efficiency counter-flow design and carry a 15-year warranty on the casing, which is molded into a unitary leak-proof structure of engineered plastic. All models are factory assembled, simple to install and nearly maintenance free. *1-800-289-3358 www.deltacooling.com sales@deltacooling.com*

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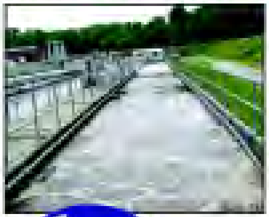

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Plast-O-Matic Valves, Inc. has developed an all-thermoplastic, air operated pinch valve with a newly designed sleeve element that lasts up to five times longer than traditional designs. The sleeve ele-ment in Series HSA is a patent-pending design featuring variable thicknesses and a tapered shape. It is molded from EPDMh2, a new formulation of Ethelyne-Propylene-Diene-Monomer using a proprietary curing process for greater elasticity, durability, and longevity. This elastomer, combined with the new design, enables the pinch element to achieve over 1,000,000 cycles under test conditions. Further Information: ***Plast-O-Matic Valves, Inc.***, *(973) 256-3000 • Fax: (973) 256-4745* ***www.plastomatic.com*** • *info@plastomatic.com*

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BUSINESS NEWS

PLANT WATCH

Dow Corning considers biomass plant to produce steam and electricity

August 20, 2010 — Dow Corning's (Midland, Mich.; www.dowcorning.com) Midland manufacturing site is considering the installation of a biomass-powered energy facility to provide a renewable, reliable and cost-effective supply of steam and electricity necessary for the site's operations. Cirque Energy, LLC, which would build, own and operate the plant for Dow Corning, recently filed for appropriate environmental permits. Biomass energy from the plant would be produced by gasification of plant-derived organic matter. The biomass energy facility at the Midland site would initially use waste wood chips, as well as dead trees or old growth harvested as part of sustainable forestry initiatives.

SNC-Lavalin receives a contract in Alberta's oil sands

August 19, 2010 — SNC-Lavalin Inc. (Montreal, Quebec, Canada; www.snclavalin.com) has been awarded an engineering and procurement contract by Grizzly Oil Sands ULC for its new Algar Lake steam assisted-gravity drainage (SAGD) facility located near Fort McMurray in Alberta. The project consists of an initial 5,000 bbl/d SAGD central-processing facility with associated well pads, flow lines, tank farm and 8-MW cogeneration facility. The detailed design of the project is expected to be completed in April 2011. This is a unique project in the fact that the execution plan is based on engineering and constructing a completely modularized processing facility capable of being trucked to site.

SGL Group expands its isostatic graphite capacity

August 19, 2010 — SGL Group (Wiesbaden, Germany; www.sglcarbon.com) will invest approximately €75 million into the capacity expansion of isostatic graphite within the next three years. This expansion supports the global growth of its customers in the solar energy, light emitting diode (LED) and semiconductor industries. A significant part of the investment will involve the construction of a new, fully automated Iso Graphite Green Production Center at the Bonn, Germany, site. Commissioning is scheduled for 2012. In this context SGL Group will increase its global, annual isostatic-graphite capacity from 5,000 metric tons (m.t.) to 15,000 m.t.

BASF plans to build dispersions plant in Huizhou, China

August 16, 2010 — BASF SE (Ludwigshafen, Germany; www.basf.com) will invest in a dispersions plant in Daya Bay Petrochemical Industrial Park in Huizhou, China. With a capacity of 100,000 ton/yr, the new plant will produce XSB dispersions for the paper industry and acrylic dispersions for industries that include coatings, construction, printing-and-packaging and adhesives. Production is scheduled to begin in the 1st Q of 2012, subject to government approval.

Outotec to design and deliver filtration technology to iron-ore project in Australia

August 5, 2010 — Outotec Oyj (Espoo, Finland; www.outotec.com) has been awarded orders for Australia's Karara Iron Ore Project, worth more than €28 million. Outotec will design, deliver and install the complete flotation circuit along with all filtration technology for the project. The Karara Iron Ore Project located in Western Australia is a 50–50 joint venture between Gindalbie Metals Ltd. and Chinese steel producer AnSteel. It is part of the development of the Karara magnetite deposit, which has the potential for more than 30-million-m.t./yr processing of magnetite over its estimated 30-year life. The first phase of operation is scheduled for 2011.

... and will deliver chromite sintering technology to South Africa

August 3, 2010 — Outotec Oyj has signed a contract with RB Met Engineering (Pty) Ltd. and Xstrata Merafe PSV for the delivery of chromite sintering technology to Xstrata Merafe's ferrochrome plant located in Rustenburg, South Africa. The contract value is approximately €17 million. The plant will treat 600,000 m.t. /yr of chromite ore. Xstrata Merafe's project is subject to an environmental permit by local authorities in Rustenburg, and the plant is expected to be operational in 2012.

BASF begins upgrade of North American catalyst manufacturing operations

August 3, 2010 — BASF Corp.'s catalyst division (Iselin, N.J.; www.catalysts.basf.com) has initiated several capital investment projects to enhance the company's manufacturing operations for copper-chrome catalysts at its production sites in Erie, Pa. and Elyria, Ohio. The multimillion-dollar investments are aimed at re-engineering, retrofitting and further automating catalyst manufacturing at both sites. BASF says the capital improvement projects will be completed in phases through the early part of 2011.

MERGERS AND ACQUISITIONS

Teijin to develop silicon inks with the acquisition of NanoGram

August 8, 2010 — Teijin Ltd. (Tokyo; www.teijin.co.jp) has acquired NanoGram Corp. (Milpitas, Calif.; www.nanogram.com) to function as a U.S. base for the development of silicon inks with semiconductor properties and the processes to manufacture such inks. As a wholly owned subsidiary of Teijin, NanoGram will pursue its interest in expanding into development of silicon-based nanoparticles, working closely with the Teijin Group's Integrative Technology Research Institute.

Air Liquide acquires a German syngas plant

August 6, 2010 — Air Liquide (Paris, France; www.airliquide.com) has signed an agreement with OXEA GmbH for the acquisition of its syngas plant based in Oberhausen, Germany. Anti-trust authorities have given their approval. This large plant has a production capacity of approximately 83,000 Nm^3/h of syngas — a mixture of hydrogen and carbon monoxide — used in the production of oxo-intermediates and oxo-derivatives, which are used in high-quality coatings, lubricants and cosmetic and pharmaceutical products.

Axens signs agreement for activated alumina business

August 4, 2010 — Axens (Rueil Malmaison, France; www.axens.net) has signed an asset sale agreement (ASA) with Canada-based Rio Tinto Alcan regarding the sale by Rio Tinto Alcan of its activated alumina business in Brockville, Ontario. The terms of the ASA are confidential. The transaction is expected to close by end-September 2010.

Bilcare enters agreement to acquire Ineos' Global Films business

August 2, 2010 — Ineos Group (Lausanne, Switzerland; www.ineos.com) has entered into a binding agreement for the sale of its Global Films business to Bilcare AG for approximately €100 million. The deal comprises the business, assets and personnel related to Ineos' Films operations located in North America, Europe and Asia. The transaction was expected to be completed at the end of August, subject to necessary regulatory filings and approvals. ■

Dorothy Lozowski

FOR ADDITIONAL NEWS AS IT DEVELOPS, PLEASE VISIT WWW.CHE.COM

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Economic Indicators

2010 2009 2008

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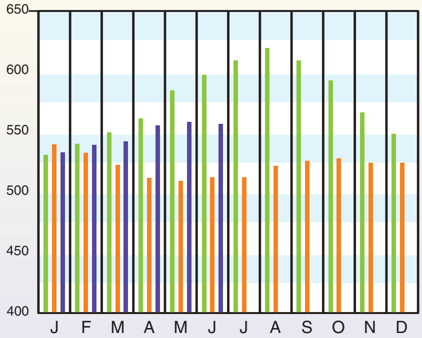
CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	June '10 Prelim.	May '10 Final	June '09 Final
***CE* Index**	556.4	558.2	512.0
Equipment	668.1	670.2	601.5
Heat exchangers & tanks	628.7	629.9	538.0
Process machinery	632.0	631.8	584.9
Pipe, valves & fittings	818.5	828.3	749.0
Process instruments	419.4	424.8	391.8
Pumps & compressors	898.4	903.1	898.9
Electrical equipment	482.2	473.2	459.8
Structural supports & misc	697.5	697.5	610.0
Construction labor	326.7	327.8	325.6
Buildings	509.4	513.9	485.7
Engineering & supervision	339.1	339.7	347.2

Annual Index:
2002 = 395.6
2003 = 402.0
2004 = 444.2
2005 = 468.2
2006 = 499.6
2007 = 525.4
2008 = 575.4
2009 = 521.9

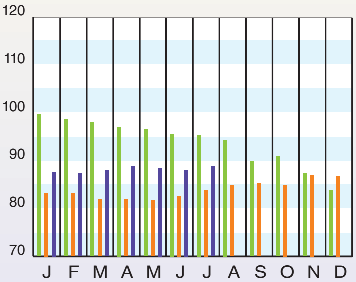


Starting with the April 2007 Final numbers, several of the data series for labor and compressors have been converted to accommodate series IDs that were discontinued by the U.S. Bureau of Labor Statistics

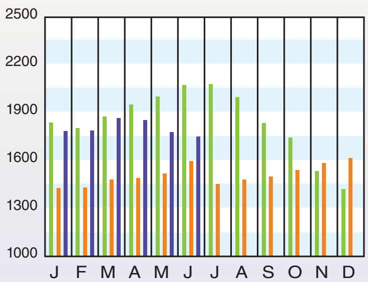
CURRENT BUSINESS INDICATORS

	LATEST		PREVIOUS				YEAR AGO	
CPI output index (2007 = 100)	Jul. '10 =	88.8	Jun. '10 =	88.1	May. '10 =	88.5	Jul. '09 =	83.9
CPI value of output, $ billions	Jun. '10 =	1,753.1	May. '10 =	1,780.7	Apr. '10 =	1,854.6	Jun. '09 =	1,597.2
CPI operating rate, %	Jul. '10 =	71.7	Jun. '10 =	71.2	May. '10 =	71.4	Jul. '09 =	66.6
Producer prices, industrial chemicals (1982 = 100)	Jul. '10 =	258.7	Jun. '10 =	267.7	May. '10 =	272.8	Jul. '09 =	236.1
Industrial Production in Manufacturing (2007=100)	Jul. '10 =	90.6	Jun. '10 =	89.6	May. '10 =	90.0	Jul. '09 =	84.1
Hourly earnings index, chemical & allied products (1992 = 100)	Jul. '10 =	152.6	Jun. '10 =	153.3	May. '10 =	152.8	Jul. '09 =	148.8
Productivity index, chemicals & allied products (1992 = 100)	Jul. '10 =	120.3	Jun. '10 =	120.6	May. '10 =	119.2	Jul. '09 =	118.5

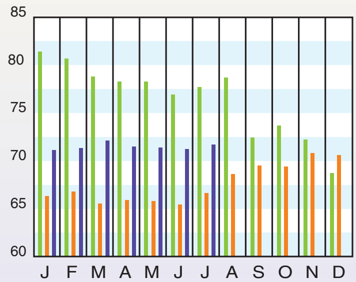
CPI OUTPUT INDEX (2007 = 100)



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



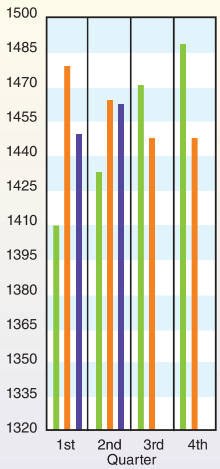
Current Business Indicators provided by Global Insight, Inc., Lexington, Mass.

MARSHALL & SWIFT EQUIPMENT COST INDEX

(1926 = 100)	2nd Q 2010	1st Q 2010	4th Q 2009	3rd Q 2009	2nd Q 2009
M & S INDEX	1,461.3	1,448.3	1,446.5	1,446.4	1,462.9
Process industries, average	1,522.1	1,510.3	1,511.9	1,515.1	1,534.2
Cement	1,519.2	1,508.1	1,508.2	1,509.7	1,532.5
Chemicals	1,493.5	1,481.8	1,483.1	1,485.8	1,504.8
Clay products	1,505.6	1,496.0	1,494.3	1,495.8	1,512.9
Glass	1,416.4	1,403.0	1,400.1	1,400.4	1,420.1
Paint	1,527.6	1,515.1	1,514.1	1,515.1	1,535.9
Paper	1,430.1	1,416.4	1,415.8	1,416.3	1,435.6
Petroleum products	1,625.9	1,615.6	1,617.6	1,625.2	1,643.5
Rubber	1,564.2	1,551.0	1,560.5	1,560.7	1,581.1
Related industries					
Electrical power	1,414.0	1,389.6	1,377.3	1,370.8	1,394.7
Mining, milling	1,569.1	1,552.1	1,548.1	1,547.6	1,562.9
Refrigeration	1,786.9	1,772.2	1,769.5	1,767.3	1,789.0
Steam power	1,488.0	1,475.0	1,470.8	1,471.4	1,490.8

Annual Index:

2002 = 1,104.2	2004 = 1,178.5	2006 = 1,302.3	2008 = 1,449.3
2003 = 1,123.6	2005 = 1,244.5	2007 = 1,373.3	2009 = 1,468.6



CURRENT TRENDS

Capital equipment prices (as reflected in the *CE* Plant Cost Index) declined in June, an unusual phenomenon given that equipment prices typically peak around August of each year.

Meanwhile, Current Business Indicators from Global Insight, Inc. show that CPI output dropped for the fourth consecutive month in June. Data from the American Chemistry Council indicate a July rebound, however, in both production and capacity utilization.

Finally, note that the bases for the CPI Output Index and Industrial Production in Manufacturing changed from 2002=100 to 2007=100 on June 25.

Visit www.che.com/pci for more. ■