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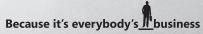


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COVER STORY

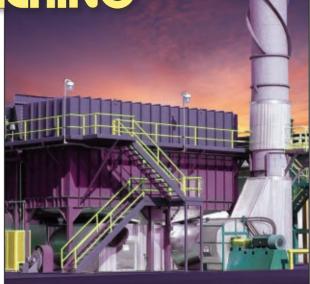
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EQUIPMENT & SERVICES

24D-1 Interphex 2010 Preview

(Domestic Edition) The 31st Interphex Conference and Exhibition will be held April 20–22, 2010 at the Jacob Javits Convention Center in New York. A sampling of products to be displayed is given, including: A valve and sensor line for hygienic processes; An ink-jet printer for heavy duty applications; A pressure gage for alternate units of measure; A weigher that improves simplicity and user ergonomics; and more

24I-2 New Products & Services

(International Edition) A benchtop FTIR for near-infrared analysis; A flowmeter that ensures reproducible chromatography; Disc valves that can handle abrasive slurries; A machinery protection solution; An air sampler for bio-aerosols monitoring; Earplugs with hybrid design; and more

52 Focus Analyzers Deposits are not a problem for this process refractometer; A gas sensor that can be calibrated remotely; Measure fluoride levels over a wide concentration range; A phosphate analyzer that consumes less reagents; Measure hydrocarbons with ppb resolution; A gas analyzer with a two-laser platform; and more

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cal Engineers The U.S. has pledged to reduce greenhouse gas emissions to 2005 levels by 2020. But the reduction target depends on the enactment of climatechange legislation in the U.S. We want to hear what you think about the issues surrounding greenhouse gas emissions. Chemical Engineering has set up a brief online survey to capture your opinions

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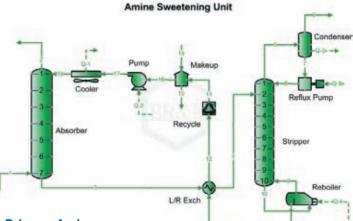
Cover Photo supplied by: Grigori A. Bunimovich



PROCESS INSIGHT

Selecting the Best Solvent for Gas Treating

Selecting the best amine/solvent for gas treating is not a trivial task. There are a number of amines available to remove contaminants such as CO_2 , H_2S and organic sulfur compounds from sour gas streams. The most commonly used amines are methanolamine (MEA), diethanolamine (DEA), and methyldiethanolamine (MDEA). Other amines include diglycolamine[®] (DGA), diisopropanolamine (DIPA), and triethanolamine (TEA). Mixtures of amines can also be used to customize or optimize the acid gas recovery. Temperature, pressure, sour gas composition, and purity requirements for the treated gas must all be considered when choosing the most appropriate amine for a given application.



Primary Amines

The primary amine MEA removes both CO_2 and H_2S from sour gas and is effective at low pressure. Depending on the conditions, MEA can remove H_2S to less than 4 ppmv while removing CO_2 to less than 100 ppmv. MEA systems generally require a reclaimer to remove degraded products from circulation. Typical solution strength ranges from 10 to 20 weight % with a maximum rich loading of 0.35 mole acid gas/mole MEA. DGA[®] is another primary amine that removes CO_2 , H_2S , COS, and mercaptans. Typical solution strengths are 50-60 weight %, which result in lower circulation rates and less energy required for stripping as compared with MEA. DGA also requires reclaiming to remove the degradation products.

Secondary Amines

The secondary amine DEA removes both CO₂ and H₂S but generally requires higher pressure than MEA to meet overhead specifications. Because DEA is a weaker amine than MEA, it requires less energy for stripping. Typical solution strength ranges from 25 to 35 weight % with a maximum rich loading of 0.35 mole/mole. DIPA is a secondary amine that exhibits some selectivity for H₂S although it is not as pronounced as for tertiary amines. DIPA also removes COS. Solutions are low in corrosion and require relatively low energy for regeneration. The most common applications for DIPA are in the ADIP[®] and SULFINOL[®] processes.

Tertiary Amines

A tertiary amine such as MDEA is often used to selectively remove H_2S , especially for cases with a high CO_2 to H_2S ratio in the sour gas. One benefit of selective absorption of H_2S is a Claus feed rich in H_2S . MDEA can remove H_2S to 4 ppm while maintaining 2% or less CO_2 in the treated gas using relatively less energy for regeneration than that for DEA. Higher weight percent amine and less CO_2 absorbed results in lower circulation rates as well. Typical solution strengths are 40-50 weight % with a maximum rich loading of 0.55 mole/mole. Because MDEA is not prone to degradation, corrosion is low and a reclaimer is unnecessary. Operating pressure can range from atmospheric, typical of tail gas treating units, to over 1,000 psia.

Mixed Solvents

In certain situations, the solvent can be "customized" to optimize the sweetening process. For example, adding a primary or secondary amine to MDEA can increase the rate of CO_2 absorption without compromising the advantages of MDEA. Another less obvious application is adding MDEA to an existing DEA unit to increase the effective weight % amine to absorb more acid gas without increasing circulation rate or reboiler duty. Many plants utilize a mixture of amine with physical solvents. SULFINOL is a licensed product from Shell Oil Products that combines an amine with a physical solvent. Advantages of this solvent are increased mercaptan pickup, lower regeneration energy, and selectivity to H_2S .

Choosing the Best Alternative

Given the wide variety of gas treating options, a process simulator that can accurately predict sweetening results is a necessity when attempting to determine the best option. ProMax[®] has been proven to accurately predict results for numerous process schemes. Additionally, ProMax can utilize a scenario tool to perform feasibility studies. The scenario tool may be used to systematically vary selected parameters in an effort to determine the



optimum operating conditions and the appropriate solvent. These studies can determine rich loading, reboiler duty, acid gas content of the sweet gas, amine losses, required circulation rate, type of amine or physical solvent, weight percent of amine, and other parameters. ProMax can model virtually any flow process or configuration including multiple columns, liquid hydrocarbon treating, and split flow processes. In addition, ProMax can accurately model caustic treating applications as well as physical solvent sweetening with solvents such as Coastal AGR[®], methanol, and NMP. For more information about ProMax and its ability to determine the appropriate solvent for a given set of conditions, contact Bryan Research & Engineering.



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Editor's Page

Opinions on greenhouse gases

little over one month ago, the United Nations Framework Convention on Climate Change (UNFCCC) received national pledges from 55 countries, to cut and limit greenhouse gas (GHG) emissions. Included in the pledges was a commitment from the U.S. to reduce its GHG emissions in the range of 17% from 2005 levels by 2020. The non-binding target came with a disclaimer, however, that hinges on an uncertainty for the world's largest emitter among developed nations: the enactment of U.S. climate legislation.

Several months ago, the prospect for U.S. climate legislation in the near term seemed much more likely than it does at CE press time. But that could change, given the mood swings that seem to have taken this issue hostage. Last month, for instance, researchers at Yale and George Mason universities released the results of a national survey in which 62% of respondents said that the U.S. should make a medium- to large-scale effort to reduce global warming, even if doing so has moderate or large economic costs. Despite a 12-point drop in that measure since the fall of 2008, most respondents — regardless of political affiliation — indicated that they support the passage of federal climate and energy policies, the survey says.

When it comes to climate change, I consider myself to be a pragmatist. I certainly don't have any black-and-white evidence that proves exactly how today's GHG concentrations or proposed legislation would play out. But here are four points that I think are often either overlooked or misunderstood:

1. Climate change and global warming are two related, but not identical terms. Climate change refers to major changes in temperature, rainfall, snow or wind patterns lasting for decades or longer. Although there are many natural sources of climate change, it is the influence of human activities on climate change that is currently under scrutiny. Meanwhile, global warming refers to an average increase in surface temperatures over time and can be considered part of climate change along with changes in precipitation, sea level and so on. The point here is that recent snow storms in Texas and Virginia do not disprove global warming. If anything, they are a nod to more extreme-weather patterns indicative of climate change.

2. The idea that CO_2 can't be considered a pollutant, simply because humans exhale it or plants consume it, takes oversimplification to an extreme. Climate change predictions aside, increasing CO_2 concentrations can — and are — contributing to increased acidity of the oceans.

3. Cap-and-trade legislation has already been proven successful in the Acid Rain program, brought to pass by the first Bush Administration in the 1990s. Crafted by an unlikely marriage of free-market conservatives and environmentalists, the program was completely successful in terms of SO₂ emissions compliance, despite some critics' concerns that it would be a way for industry to buy its way out of fixing the problem. Meanwhile, other critics claimed that the limits on pollution would cause electricity bills to rise, but electricity rates are lower now (in constant dollars) than they were in 1990.

4. Natural gas, while cleaner burning and less carbon intensive than coal, is a key chemical feedstock. Further shifts toward natural gas as an energy source would have economic impacts for industry and consumers alike.

While these and other nuances are hammered out in the U.S. regulatory system, the combination of mandatory GHG reporting and increasing scrutiny in the financial sector could have its own influence (see p. 17 for more). In the meantime, we want to hear your opinions on GHG regulation, climate change and everything in between. Please visit our Website (www.che.com) for a brief online survey that will be open until April 10. Rebekkah Marshall



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Letters

ChemInnovations Call for Papers

Chemical Engineering has recently announced the launch of ChemInnovations 2010 (www.cpievent. com), a new conference and exhibition for the chemical process industries (CPI) to be held October 19–21 at Reliant Park (Houston), in partnership with TradeFair Group (Houston, www.tradefairgroup.com). The event is specifically focused on presenting the innovative technologies and approaches that are vital to addressing today's processing challenges, while helping attendees anticipate market and regulatory trends in the CPI.

The 2010 ChemInnovations conference is issuing an industries-wide call for papers. Prospective authors are invited to submit a 200–300 word abstract for consideration by the advisory committee. Abstracts should focus on innovative, practical and proven approaches to the CPI's biggest challenges. Abstracts of a how-to orientation will be given preferential consideration. Suggested topics include, but are not limited to, the following:

Process intensification and optimization Aging plants: Shuttering, revamping and converting Feedstock flexibility Energy efficiency and flexibility Cost estimation

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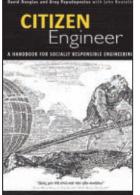
Submission details

- Submit a 200–300 word abstract via email to Kim Arellano, conference director: kima@tradefairgroup.com
- Abstract deadline: Friday, March 26, 2010.

You will be notified 60 days after the submission deadline on whether or not your abstract was accepted. If your abstract is accepted, you must register for the event within 14 days of notification at the speaker registration rate of \$225. ■

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Bookshelf



Citizen Engineer: A Handbook for Socially Responsible Engineering. By David Douglas, Greg Papadopoulos and John Boutelle. Prentice Hall, One Lake Street, Upper Saddle River, NJ 07458. Web: www.prenhall.com. 2009. 292 pages. \$27.00.

Reviewed by Arthur Schwartz, National Society of Professional Engineers, Alexandria, Va.

Numerous works about the professional practice of engineering have been written that touch on ethics, law, contracts, intellectual property, risk management, techology and other important issues relevant to all engineering disciplines. Many of these books have tended to focus either on engineering private practice (consulting, for example) or on a narrow engineering-related field, and have failed to frame the key issues affecting engineers working in industrial and manufacturing settings.

David Douglas and Greg Papadopoulos recently collaborated on an impressive and ambitous work that is strikingly different than previous books. In "Citizen Engineer," the authors issue a "clarion call" to engineers, particularly software and chemical engineers. The authors assert that engineers must understand and embrace a new role as society's movers, shakers and leaders. While the basic theme of the book is not new, the authors appear to be calling for a rebirth of engineering as a profession.

"The traditional role of engineers has changed," say the authors, and engineers need to become the masters of the post-technical present and future. They write, "... engineering is no longer concerned with finding a simple, elegant way to implement a set of design requirements... we need knowledge of subjects well beyond the scope of traditional engineering. A successful engineer needs to be part environmentalist, part intellectual property attorney, part business executive, and part diplomat — not to mention an expert in an engineering discipline, a great teammate and a skilled communicator."

The authors posit that several recent trends are further redefining the role of the engineer in society. For instance, the increasing complexity of products leads to greater dependence upon engineering; yet understanding of engineering and its underlying sciences is relatively low. This can lead, the authors note, to poor public policy and misconceptions that hinder innovation. The authors urge engineers to take a more proactive role in engaging, communicating with and leading society.

The book mostly explores modern engineering and provides practical guidance on topics of increasing interest and urgency to engineers — particularly environmental considerations of product design, intellectual property and contractual issues. The book examines how eco-effective, technoresponsible products and services can translate into new

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opportunities for businesses and accelerated career paths for engineers. It contains excellent advice for students and recent graduates beginning engineering careers.

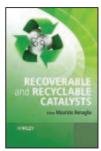
While the book is highly impressive, its emphasis on the social responsibilities of engineers precludes mention of the important role played by engineering licensure in protecting public health and safety — an oversight. Also, the book could have acknowledged a conversation currently underway in the profession regarding the need for meaningful additional education to support professional practice — a point that would have added credibility to the authors' central thrust.

Overall, the authors have commendably highlighted the need for engineers to incorporate social responsibility into their profession.



Giant Molecules: From Nylon to Nanotubes. By Walter Gratzer. Oxford University Press, 198 Madison Ave., New York, NY 10016. Web: www.oup. com. 2009. 254 pages. \$24.95.

Computational Techniques for Multiphase Flows. By Guan Heng Yeoh and Jiyuan Tu. Butterworth-Heinemann, 30 Corporate Drive, Suite 400, Burlington, MA 01803. Web: www. elsevier.com. 2009. 664 pages. \$130.00.



Recoverable and Recyclable Catalysts. Edited by Maurizio Benaglia. John Wiley and Sons Inc., 111 River Street, Hoboken, NJ 07030. Web. www. wiley.com. 2009. 500 pages. \$160.00.

High Energy Density Lithium Batteries: Materials, Engineering and Applications. Edited by Katerina E. Aifantis, Stephen A. Hackney and R. Vasant Kumar. John Wiley and Sons Inc., 111 River Street, Hoboken, NJ 07030, Web. www.wiley.com. 2010. 296 pages. price to be determined.



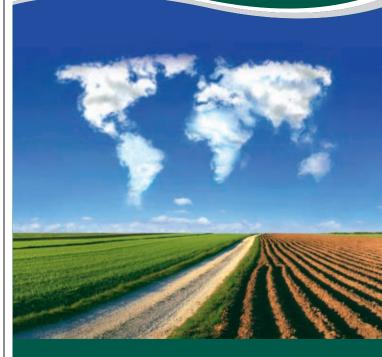
Food Stabilisers, Thickeners and Gelling Agents. Edited by Alan Imeson. John Wiley and Sons Inc., 111 River Street, Hoboken, NJ 07030, Web. www.wiley.com. 2009. 368 pages. \$199.99.



Materials and Design: The Art and Science of Material Selection in Product Design. By Michael F. Ashby and Kara Johnson. Butterworth-Heinemann, 225 Wildwood Ave., Woburn, MA 01801. Web: www.elsevier.com. 2009. 344 pages. \$59.95. ■

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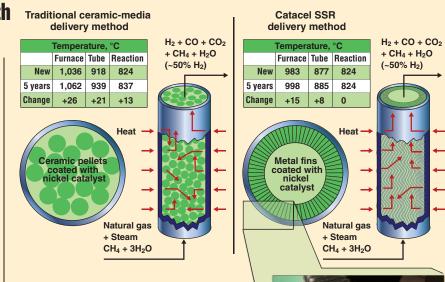
Edited by Gerald Ondrey

Slash energy consumption with this steam reformer reactor

Steam reforming of methane into hydro-gen takes place in catalyst-packed alloy tubes that are heated in a furnace. Up to now, this energy-intensive reaction has used catalyst-impregnated ceramic pellets, which are poured into the tubes. However, these ceramic pellets do not provide homogeneous heat transfer, which compromises reaction efficiency, and they are prone to crushing, which degrades the catalyst and thus necessitates change-out every 3-5 yr. Now, an alternative catalyst support that provides 2.5 times the surface area, 1.3–1.6 times greater heat-transfer rates, and lasts at least two times longer than ceramic supports has been commercialized by Catacel Corp. (Garrettsville, Ohio; www.catacel.com).

Tested in cooperation with the NASA Glenn Research Center (Cleveland, Ohio; www.nasa.gov/centers/glenn), Catacel's patented Stackable Structural Reactor (SSR) consists of metal foil with flow channels formed onto the surface. The catalyst is bonded to the foil, which is then assembled into canisters that can be stacked vertically into reformer tubes.

SSR's improved heat-transfer capability enables the furnace to operate at 40–50°C lower temperatures than if the reformer tubes were packed with ceramic pellets,



thereby saving about 10% of the fuel needed to fire the furnace, says Bill Whittenberger, president and founder of Catacel. Alternatively, operating a reformer with SSR at the same (higher) temperature as ceramic media can boost the H₂ production capacity by 25–35% in the same reformer, he says.

Test results for SSR were subsequently validated with the first commercial demonstration of the technology — a $250\text{-m}^3/\text{h}$ H₂ plant in Europe that has been in continuous operation since July 2008. Catacel is now negotiating with potential users to implement or license the technology.

A new approach to flexible energy converting material

Aresearch team at Princeton University (Princeton, N.J.; www.princeton.edu) led by Michael McAlpine and Yi Qi has devised a process for integrating nanoscale piezoelectric ribbons into flexible rubbers, enabling development of flexible, energy-harvesting materials. Efficient, flexible energy-conversion materials could be used as wearable energy-harvesting systems for mobile electronic devices or implantable medical devices. Piezoelectric materials become electrically polarized when subjected to mechanical stresses.

The team's approach involved depositing 500 nm-thick, micrometer-wide crystalline ribbons of the piezoelectric material lead zirconate titanate (PZT) on magnesium oxide wafers, then separating the PZT material from the MgO with an etching process. The ribbons are then transferred onto a polydimethylsiloxane (PDMS) rubber substrate by bringing a layer of PDMS into contact with the PZT ribbons still on the MgO wafer. The PDMS layer is then peeled back to retrieve the ribbons, resulting in a "piezo-rubber" chip that contains an array of the piezoelectric strips.

Subsequent studies of the piezo-rubber chips confirm that the piezoelectric performance of the PZT ribbons is retained after transferring to the rubber substrates. The researchers hope to scale up the process to produce larger sheets of the material, and to further study the mechanics of piezoelectric materials on stretchable platforms.

A new Kraft pulp

Weyerhaeuser Co. (Federal Way, Wash.; www.weyerhaeuser.com) says it has begun commercial production of a new grade of Kraft pulp for the cellulose derivatives market. Cellulose derivatives are used in various products, including lacquers, paints, inks and thickening agents.

March 2010

The new pulp, called Pearl429, "is essentially a new class of softwood Kraft pulp," says Don Atkinson, vicepresident of marketing and new product development for Weyerhaeuser Cellulose Fibers. The company declines to give details on the production process, except to say that it is a continuous Kraft process, whereas many other specialty pulps are made by a batch process. The new method is said to generate higher yields than typical dissolving pulp manufacturing processes, from the same amount of raw materials, with better product uniformity. Weyerhaeuser adds that

(Continues on p. 12)





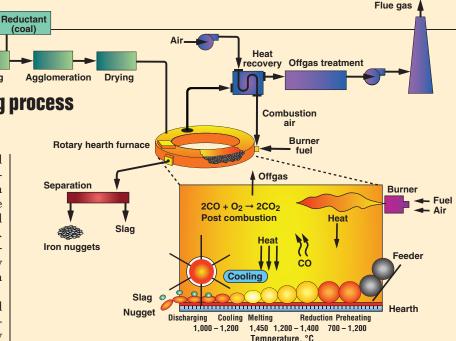
Iron

oxide

Mixing

n January, the world's first commercial plant to use the ITmk3 process began production of iron nuggets, which are used in steelmaking. The plant, constructed by Kobe Steel, Ltd. (Tokyo; www.kobelcom) and Steel Dynamics, Inc. (Fort Wayne, Ind.; www. steeldynamics.com) at Hoyt Lakes, Minnesota, is expected to reach its design capacity of 500,000 metric tons per year (m.t./yr) in mid 2010.

Developed by Kobe Steel, ITmk3 is said to be the next-generation ironmaking process (CE, January 2002, p. 15), and is totally different from the traditional blast furnace method. The process evolved from Fastmet (CE, March 1995, pp. 37-41), developed by Midrex Technologies, Inc. (Charlotte, N.C.; www.midrex.com) and Kobe Steel (Midrex's parent company). Both Fastmet and ITmk3 use a rotary hearth furnace (RHF), a large turntable that rotates within a doughnutshaped chamber. Feed pellets — agglomerates made from iron-ore fines and pulverized coal - are charged into the hearth (1–2 layers deep) and are heated by burners firing from above and by the combustion of gases released from the reduced pellets. One revolution of the hearth takes about 10 min. In Fastmet, the product is direct reduced iron, but in ITmk3, the pellets are melted in the last zone of the hearth to produce iron



nuggets of the same quality as pig iron, with slag as a by-product. Heat is also recovered from the offgas to preheat combustion air.

ITmk3 can use lower-cost iron-ore fines and steaming coal, which are difficult to use in blast furnace ironmaking, says Kobe Steel. Unlike blast furnace operations, ITmk3 eliminates the need for raw material pretreatment, such as coke ovens, sintering plants and pellet plants. ITmk3 iron nuggets can be produced in just 10 min, whereas blastfurnace pig iron can take up to 8 h. For steelmakers, the high-grade nuggets improve the productivity and energy efficieny of electric arc furnaces. The process is also highly suitable for mining sites and can be profitable even for small mines adds the firm.

Nanofiber cartridge filters achieve 0.03-mm rating at high flow and low pressure

A 1.5-mm-thick layer of a new filter material based on nanoscale alumina has shown the ability to filter greater than 99% of 0.03-mm latex spheres or 0.025-mm MS2 viruses with sustainable water velocities of around 1.5 cm/s at pressures of 0.7 bar. Developed by the Argonide Corp. (Sanford, Fla.; www. argonide.com), the alumina nanofibers have a surface area of 350-500 m²/g and form the active component in a non-woven filter matrix with a 2-mm pore size. The filter medium works on the principle of electrostatic adsorption, where the positively charged filter media attract and adsorb negatively charged particles, such as pathogens and biological macromolecules.

"Significant effort led to the successful commercialization of an electropositive, fibrous, pleated-depth filter that can substitute for the asbestos filters that were phased out years ago," Argonide president Fred Tepper explains. "We have a 'game-changing' product here," adds Tepper.

The nanoscale fibers — principally boehmite (Al-O-OH) — are 2 nm in diameter and range in length from tens to hundreds of nanometers. The nanoscale alumina is distributed over a microglass fiber matrix that is modified with cellulose to increase the strength and flexibility of the matrix. At a nanoalumina content over 15 wt.%, the filter media are highly electropositive,

(Continued from p. 11)

Pearl429's characteristics allow it to be used in various applications that use cellulose esters and ethers.

Bioacrylic milestone

Since beginning pilot-scale development six months ago, OPX Biotechnologies, Inc. (Boulder, Col.; www.opxbiotechnologies. com) has reduced bioacrylic production cost by 85% towards the commercial target of 50¢/lb. The company uses its Efficient Directed Genome Engineering (Edge) technology to develop microbes and processes for making chemicals. A demonstration plant is planned for 2011, and a full-scale commercial plant in 2013.

and a 1.5-mm-thick layer (as in the pleated cartridge) is capable of adsorbing >99.9999% of the 25-nm-sized MS2 virus, the company says. In recent testing, the cartridges tolerated high dirt loads, salt concentrations (200 g/L) and alkalinity (pH>9.5).

Argonide's cartridges, marketed under the name NanoCeram, will be presented at the American Filtration and Separations Society Annual Meeting (San Antonio, Tex.; March 22–25). NanoCeram filters are now in use for applications such as filtering potable water, protecting reverse osmosis membranes, for a clean-in-place process and for iron and copper removal in chillwater systems.

Conductive fibers

Teijin Ltd. (Tokyo and Osaka; www. teijin.co.jp) and the Tokyo Institute of Technology (both Japan; www.titech. ac.jp) have developed a highly crystalline carbon nanotube fiber (CNF) that has a 30% higher electrical conductivity than conventional fibers. The highly conductive CNFs are made using a conventional melt-spinning process, and no catalyst is required, which leads to high-purity fibers 20 µm long and 100–300-nm dia. Teijin plans to commercialize the new CNF in 2011, with potential applications in lithium-ion batteries, electrodes and additives for secondary batteries, plastic additives, fuel cells and gasdiffusion layers.

The technique achieved removal efficiencies of 85–92% for lead ions after 2–3 wk operation. However, the removal efficiency of chromium ions has thus far been below 18%, due to chromium's stronger adsorption to soil particles.

Ottosen believes the hybrid system can be further developed as a continuous, *ex-situ* remediation process, which can be combined with soil washing.

Combined membrane separation and electrokinetics speeds soil remediation

Toxic heavy metals can be drawn out of soils far more quickly than the traditional methods using a process developed by a team from the Technical University of Denmark (DTU; Lyngby; www.dtu.dk) and the Universidad Técnica Federico Santa María (Valparaíso, Chile; www.usm.cl).

Conventional electrokinetic methods apply a strong d.c. electric field to cause ions of heavy metals, such as cadmium, copper, zinc, lead and chromium, to migrate through the soil. However, this process can take months to achieve adequate cleanup.

The team combined traditional electrokinetic soil remediation with conventional electrodialysis, resulting in a faster and more thorough way of cleaning soils contaminated with heavy metals. DTU's team member Lisbeth Ottosen explains that, in the new setup, soil is treated in suspension, and ion exchange membranes separate the soil suspension and the processing solutions at the electrodes. The addition of ion exchange membranes ensures the main direction for the electromigration within the contaminated soil is out of the soil.

Laboratory experiments were conducted in cylindrical PMMA (polymethyl methacrylate) partitioned into a central compartment and two electrode compartments. An anion exchange membrane separates one electrode compartment from the central compartment, and a cation exchange membrane separates the central compartment from the other electrode compartment. An overhead stirrer is used to keep contaminated soil in suspension, and the pH in the electrode compartment is maintained at around two.

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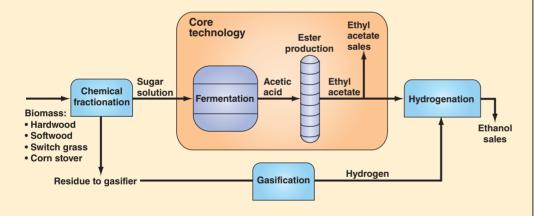
Metathesis catalysts

Ethanol and other chemicals from biomass

The ethanol yield from biomass has been increased by about 50% over conventional yields in a process developed by ZeaChem Inc. (Lakewood, Colo.; www.zeachem.com). ZeaChem has tested the process (diagram) in a 3,500-gal fermenter and will activate a demonstration plant to process tree residues in Boardman, Ore., in late 2010. The plant will produce 250,000-gal/yr of either ethanol or ethyl acetate, says Jim Imbler, president. ZeaChem plans to start up a 25-million-gal/ yr commercial plant at the site in 2013 and ultimately expects to produce ethanol for less than \$1/gal.

Cellulosic biomass is treated by acid hydrolysis and the resultant aqueous solution of glucose and xylose is fermented by an acetogen, a naturally occurring bacterium that converts the sugars to acetic acid. The acid is esterified to obtain ethyl acetate, all or part of which is hydrogenated to produce ethanol. Hydrogen is obtained by gasifying lignin residue from the acid hydrolysis process. This is distinct from conventional biomass processes, in which ethanol is produced by yeast in the fermentation step. Imbler explains that yeast fermentation creates one molecule of CO_2 for every molecule of ethanol, whereas the acetogenic method produces no CO_2 . The combination of acetic acid and H_2 production achieves a net energy value (NEV) nearly ten times that of the conventional route, he says.

In a related development, ZeaChem has produced glacial acetic acid (>99% purity), used in a wide range of products. The acid is concentrated by using a commercial solvent to extract it from the fermentation broth, then separating and recycling the solvent. Solvent extraction uses only about 25% as much energy as the conventional distillation method, says Imbler.



Furnace tube coatings reduce carbon formation and increase efficiency in olefins plants

Coating technology that essentially eliminates carbon buildup on the interior of steam cracker tubes will be commercialized by Quantiam Technologies Inc. (Edmonton, Alta; www. quantiam.com). Commercial-scale tests in five ethylene crackers indicate that the coating can extend the time between furnace decokings to 1–2 yr for light feedstocks, whereas uncoated tubes have to be decoked about every 30 days, says Steve Petrone, chief executive officer.

Petrone notes that there are two main sources of carbon buildup in furnace tubes: filamentous coke, whose formation is catalyzed at high temperatures from the nickel and iron in the steel tubes; and amorphous coke that deposits from cracking the gaseous hydrocarbon feed. Quantiam's coating prevents the former by shutting down the coke-forming mechanism. The accumulation of gas-phase coke deposits is prevented by a catalyst in the coating that converts the coke to CO and CO₂. Petrone declines to give details on the coating or catalyst, except to say the coating is a composite consisting of a metal matrix with ceramic and intermetallic components.

Petrone says the coating is stable at tem-(Continues on p. 16) The Catalyst Business Line of Evonik Industries AG (Essen, Germany; www.evonik.com) has launched three new homogeneous catalysts that cover a broad range of reactions in cross metathesis, ring-closing metathesis and ring-opening metathesis. Metathesis is a reaction used for developing and producing advanced plastics, or active ingredients for pharmaceuticals and pesticides. The total cost per kilogram of the new catalysts — catMETium RF2. RF3 and RF4 — includes the license fees for the use of intellectual property rights (RF = royalty free), so customers can use the catalysts without limitatons, savs the firm.

Cellulosic biofuels

Also launched at the National Ethanol Conference (see also the story on p. 16) is Accellerase DUET, the latest generation of Genencor's (Palo Alto, Calif.; www.genencor.com) enzymes used to convert biomass into fermentable sugars. With improved overall hemicellulase activity, Accellerase DUET builds on the advances in beta-glucosidase and cellular activity of its predecessor (Accellerase 1500), enabling DUET to achieve higher sugar and biofuel yields - often at a three-fold lower dosing, says the firm.

Bio-based adipic acid

Verdezyne (Carlsbad, Calif.; www.verdezyne.com) has achieved a proof of concept in its development program by demonstrating production and recovery of adipic acid made by a veast microorganism from an alkane feedstock. This is the company's first milestone towards demonstrating an entirely feedstock-flexible (plant derived sugar, oils or alkanes) fermentation process for making adipic acid — an important starting material for making polyamides and polyurethanes.

Verdezyne estimates that its route to adipic acid has at least a 20% cost advantage over the traditional petroleum-based route. The company plans to partner for scaleup demonstration in the next year.

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CHEMENTATOR

FURNACE TUBE COATINGS

(Continued from p. 14)

peratures up to 1,130°C and has tolerated sulfur levels up to 3,000 ppm. By avoiding carbon buildup, the coating can also lower energy costs by 3-10% and increases product throughput by allowing a reduction in steam use, since one function of the steam is to oxidize tube surfaces to protect against coke formation.

Quantiam's pilot plant capacity is about 1 million (internal) in.²/yr, or enough for two commercial furnaces. Aided by an investment of approximately \$3 million each from BASF Venture Capital GmbH (Ludwigshafen, Germany) and Ursataur Capital Management L.P. (Toronto, Ont.), the company plans to scale up to 3 million in.²/yr by April 2011. Petrone says the payback time from using the coated tubes will be less than a year.

The cost of producing bioethanol takes a nosedive

ast month at the National Ethanol Conference at Orlando, Fla., Novozymes' A/S (Bagsvaerd, Denmark; www.novozymes.com) president and CEO, Steen Riisgaard, launched Cellic CTec2, a new enzyme product enabling the biofuel industry to produce cellulosic ethanol at a price below \$2/gal - comparable to the current cost of gasoline and conventional ethanol in the U.S. Cellec CTec2 has a higher potency (a factor of two higher than its predecessor) for breaking down agricultural waste (such as corn cobs and stover and sugarcane bagasse) into fermentable sugars. Compared to existing enzymes, CTec2 can reduce enzyme dosing by 50%, thereby reducing the ethanol production costs by 50¢/gal, says Riisgaard. This eliminates the need — and capital expense — for onsite production of the enzyme at large ethanol plants. Delivery of two truckloads per week is enough for a large-scale ethanol plant, which is comparable to the enzyme-delivery needs at a conventional starch-based ethanol plant, he says.

CTec2 is a mixture of "more than a handful" of different enzymes that work together to break down cellulose into fermentable sugars. The enzymes - both cellulases and hemicellulases - are extracted from various fungi, and the most efficient then cloned into a microoganism (Tricoderma) and expressed by fermentation. The enzymes are concentrated and purified into a liquid product.

Riisgaard says the company is building a new production facility in Blair, Neb. (startup early 2012) to serve the North American market, and already has sufficient production capacity to serve markets in the EU and China. Currently, the outlet for bioethanol for fuel - regardless if the ethanol is from grains or waste — is limited by the current E10 blend restriction (a limit of 10% ethanol in gasoline) set by the U.S. Environmental Protection Agency (EPA; Washington, D.C.). Nevertheless, Riisgaard says that there is enough agricultural waste already generated to supply up to 25% of the world's gasoline requirements, and this fraction will only increase as enzymes are developed to handle other cellulosecontaining materials, such as wood chips or weeds that can be grown on fallow land.



GREENHOUSE GASES: U.S. STARTS COUNTING

From emissions estimates to related financial risks and opportunities, the CPI are going to add it up

s 2009 came to a close, the U.S. crossed a key milestone in the path toward regulating greenhouse gas (GHG) emissions. Nearly 10,000 facilities (Figures 1 and 2) — a significant portion of them in the chemical process industries (CPI) - became subject to the U.S. Environmental Protection Agency's (EPA; www.epa. gov; Washington D.C.) Final Mandatory Greenhouse Gases Reporting Rule. The rule requires that applicable facilities begin collecting data on January 1. 2010 for annual GHG emission reports that are due to EPA by March 31, 2011. Although the rule itself does not limit GHG emissions, the collected data will be used to inform future climate-change policies and programs, EPA says.

Whether it is because of the relatively quick pace that this rule took in being made official or the extremely loud noise from broader GHG debates in the mainstream media, many chemical engineers found themselves in a year-end rush to prepare for the January 1 milestone. Others have requested extensions, which will expire at the end of this month. Meanwhile, for all facilities that are subject to the rule, the next deadline for preparing a formal monitoring plan is right around the corner on April 1.

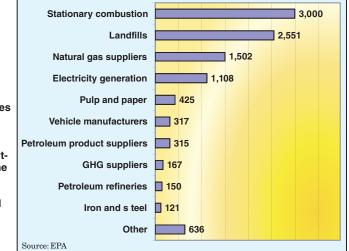
As the U.S. CPI grapple with the specifics of EPA's reporting rule, curiosity is building globally around the extent to which GHG reduction initiatives will be in demand. For now, the future of U.S. climate policy is caught in a storm of political and social debates, clouding the picture at the regulatory level FIGURE 1. The EPA estimates that 10,000 U.S. facilities will be covered by the mandatory reporting rule that came into effect on January 1. The category defined as "Other" is detailed in Figure 2

(for more, see p. 5 where we ask you to weigh in with your own opinions). Nevertheless, GHG regulation is already a reality in other parts of the world, and a clear motivation for GHG reductions is emerging through the U.S. financial sector. Recent moves to increase transparency into a given company's GHG risks and opportunities financially could provide the ultimate incentive for the CPI's investment into technologies that help reduce so-called carbon footprints. After all, financial motivation is primarily what has been behind most of the CPI's GHG reductions thus far.

Key aspects of the rule

In general, EPA's GHG reporting rule (www.epa.gov/climatechange/ emissions/ghgrulemaking.html) defines applicability and requirements for stationary combustion sources, 20 chemical process categories, and more (see the box, p. 19). For most sources, the reporting threshold is 25,000 metric tons per year (m.t./yr) CO_2 equivalent (CO_2e). The gases that must be reported are CO_2 , CH_4 , N_2O and fluorinated GHGs, which





include HFCs (hydrofluorocarbons), PFCs (perfluorocarbons) and SF₆ (sulfur hexafluoride). Each facility must evaluate which part (or parts) of the rule apply. For example, a large petroleum refinery with cogeneration could conceivably be subject to all three of the following subparts: stationary combustion, petroleum refining and petroleum product suppliers.

Within each subcategory, reporting requirements are divided into four tiers, which define whether the data should be calculated or measured by instrumentation and methods for doing so. "Tier 1 is the easiest to measure but the least accurate," explains Terry Moore, principal at Carbon Shrinks LLC (Austin, Tex.; www.carbonshrinks. com), while "Tier 4 is the most complex and expensive to measure but the most accurate." Since tiers are generally aligned according to the size of the unit, Moore says, a single facility could be directed to use different tiers for different combustion or industrial process units. Meanwhile, a facility may elect to use the methods of a higher tier than is applicable, but not a lower one.

Measurement methods apply not only to direct measurement of GHG emissions, says Moore, but to other data that must be reported for a given industrial process and to data that are required for the calculations. Necessary data can include fuel used, high heat value of fuels, organic carbon content of raw materials and so on. EPA will verify emissions data as opposed to involving third parties.

Areas of ambiguity

In assessing how to meet the requirements of the mandatory GHG reporting rule, chemical processors have encountered several areas of ambiguity. One of those areas involves the use of existing O_2 monitors in calculating CO_2 emissions. "This is allowed in California's GHG rule, for example, but not allowed under the EPA rule," says Barney Racine, software development manager at Honeywell Process Solutions' (Phoenix; www.honeywell.com/ ps) Environmental Solutions Group.

Another area of uncertainty is calibration. EPA says that flowmeters and other monitoring equipment need to be calibrated to meet 5% accuracy requirements prior to April 1, 2010. In a document entitled "Special Provisions for 2010" and issued in January, however, EPA qualified that requirement by saying that initial calibration may be postponed after April 1 in two cases. The first exception describes monitoring equipment that has already been calibrated according to a method specified in the applicable subpart of the rule and for which the previous calibration is still active. In this case, the instrument does not need to be recalibrated until the previous calibration has elapsed. The second exception is for units that operate continuously with infrequent outages and in which calibration would require removing the device from service, thereby disrupting process operation. In this case, initial calibration may be postponed until the next scheduled maintenance outage.

Recalibration, itself, has also come under question because the EPA rule refers to a minimum recalibration frequency while also alluding to the instrument manufacturer's specification. "If the manufacturer's frequency is different than EPA's, the lesser of the two applies," explains Allen Kugi, application engineer at Fluid Components International (San Marcos, Calif.; www.fluidcomponents.com).

Possibly the biggest issue or controversy, discussed recently at the National Petroleum Refiners's Assn. (NPRA; Washington, D.C.; www.npra. org) GHG Conference in Houston, was a ruling that flowmeters had to be temperatureand-pressure compensated with instruments located at the flowmeter, rather than from other process areas, says Chris Jones, Green Initiative marketing leader at Honeywell Process Solutions. "This provides an enormous challenge for most companies, as they do not have temperature/

pressure instrumentation at every flowmeter." At the meeting, the EPA stated that it "heard the outcry" and would reevaluate its decision, Racine says.

Timeline and the next steps

In every subpart that identifies specific measurement methods that require instrumentation, affected facilities must comply by installing or upgrading instrumentation if it doesn't meet specifications. Timing on such upgrades differs for two cases, explains Carbon Shrinks' Moore:

- 1. CEMS upgrades: Facilities required to use Tier 4 have until January 1, 2011 to get their continuous-emissions-monitoring-systems (CEMS) upgrades installed and certified
- 2. Other instrument upgrades: Facilities may use "best available measurement methods" in lieu of required instrumentation until March 31, 2010. After that date, they must either use the required instrumentation or receive an extension from EPA, but the final date to request extensions was January 31, 2010

Beyond that, the next major deadline is for completion of a monitoring plan, which is required to be in place at each reporting facility by April 1. Since the purpose of the monitoring plan is to document the process and procedures for collecting and reviewing the data needed to estimate annual GHG emis-

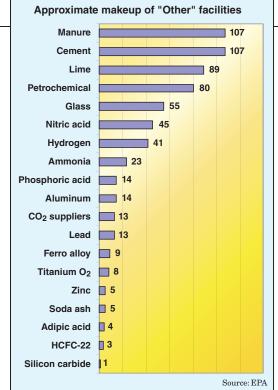


FIGURE 2. The category defined as "Other" in Figure 1 is detailed here

sions, EPA says, the plan needs to be in place prior to collecting data to ensure consistency and accuracy. EPA further emphasizes that the plan does not have to be complex and can rely on existing corporate documents like standard operating procedures (SOPs) and monitoring plans developed for compliance with other air programs. Even facilities that have been granted an extension to use best available methods to estimate GHG emissions for a period beyond April 1, 2010, are required to have a plan developed for the basic procedures that will be used to collect data. As a facility's data collection methods change and evolve, the monitoring plan must be revised to reflect the changes.

EPA says it intends to have the electronic reporting system operational in January 2011, approximately three months in advance of the March 31, 2011, reporting deadline. EPA intends to make training on the emissions reporting system available in fall 2010 and continuing into 2011. The electronic reporting system will include a separate module for registering users and facilities, scheduled to be operational and ready for training by summer 2010.

Collateral impact

Even though the expressed intent of EPA's GHG reporting rule is to inform

WHAT IS COVERED?		
The U.S. EPA's Mandatory Greenhouse Gas Reporting Rule is divided into 25 source categories and 5 types of sup- pliers of fuel and industrial GHGs. It is im- portant to recognize that any facility can be subject to multiple source categories.	 Phosphoric acid production (Subpart Z) Silicon carbide production (Subpart BB) Soda ash production (Subpart CC) Titanium dioxide production (Subpart EE) Municipal solid waste landfills** (Subpart HH) Manure management systems** (Sub- 	 Miscellaneous uses of carbonate (Sub- part U) A number of source categories have been postponed. EPA plans to further review public comments and other information before deciding on whether or not to in- clude them in future versions of the rule.
"All-in" source categories	part JJ)	
A facility is subject to the relevant subpart		Suppliers
of the rule for any of the following source	Threshold categories	All producers of the following are required
categories that exist within its boundaries:	These categories are aggregated together	to report the quantity of each product
 Electricity generation (Subpart D) Adipic acid production (Subpart E) 	to evaluate the 25,000 m.t. CO ₂ e per year reporting threshold:	introduced into the economy and GHG emissions associated with 100% oxidation
Adipic dela production (Subpart E) Aluminum production (Subpart F)	• Stationary fuel combustion (Subpart C)	of fuels and 100% release of gases:
• Ammonia manufacturing (Subpart G)	• Ferroalloy production (Subpart K)	• Coal-based liquid fuels (Subpart LL)
• Cement production (Subpart H)	• Glass production (Subpart N)	Petroleum products (Subpart MM)
 HCFC-22 production and HFC-23 de- struction* (Subpart O) 	 Hydrogen production (Subpart P) Iron and steel production (Subpart Q) 	 Natural gas and natural gas liquids, in- cluding all fractionators and local gas
 Lime manufacturing (Subpart S) 	 Lead production (Subpart R) 	distribution companies (Subpart NN)
 Nitric acid production (Subpart V) 	• Pulp and paper manufacturing (Subpart	 Industrial greenhouse gases (Subpart
 Petrochemical production (Subpart X) 	AA)	00)
 Petroleum refineries (Subpart Y) 	 Zinc production (Subpart GG) 	 Carbon dioxide (Subpart PP)

* Processes that are not co-located with a HCFC-22 production facility and that destroy more than 2.14 metric tons of HFC-23 per year **That emit 25,000 m.t. CO₂ e or more per year

public policy, the results could very easily have broader implications. "If EPA publishes, say, industry-average GHG-intensity numbers for pulpand-paper facilities of x m.t. CO₂e per ton of paper manufactured, some big paper customers may use that to set a procurement policy of y m.t. CO₂e per ton of paper as their minimum standard," says Carbon Shrinks' Moore. "In general this would reward more efficient plants and penalize less ef-

ficient ones, as well as create a new type of business case for investment in reducing GHG emissions for future annual reports to the EPA."

Already, one new index aims to achieve a similar result sooner. Last

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WHAT IS COVERED?

month, ECPI, a Milan-based research, ratings and indices company, announced the launch of its Global Carbon Equity Index. Developed in partnership with Arthur D. Little, a global management consulting firm, the new index aims to highlight 40 companies best equipped to prosper in a tougher climate-legislation environment. "The ECPI Global Carbon Equity Index has proven to outperform the market in both bull and bear markets, even through one of the worst recessions in history," says Paolo Sardi, CEO of ECPI Luxembourg. "Regular outperformance will not only provide investors with financial security but help dispel the myth that sustainable investment issues are only a concern

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for investors in strong, non-turbulent market conditions." Carbon-intensive sectors will be selected annually based on available carbon emissions data. Akzo Nobel BV, Johnson & Johnson, Eni S.p.A. and VALE SA are some of the top performers from the CPI that make up the index this year.

Over the next year, as the U.S. CPI begin to collect GHG emissions data, the connection between carbon intensity and financial performance will become more visible for any company that is publicly traded in the U.S. On January 27th, the U.S. Securities and Exchange Commission (SEC) issued new interpretive guidance on existing SEC requirements to clarify what publicly traded companies need to disclose to investors in terms of climaterelated "material" effects on business operations. The guidance specifically highlights impact of legislation and regulation; impact of international accords; indirect consequences of regulation or business trends; and physical impacts of climate change.

Increased investor scrutiny and any prospect of GHG regulations would influence how the CPI approach GHG reporting in the future. For now, most reporters "are adopting a wait-and-see attitude, and just meeting the minimum reporting requirements," says Honeywell's Racine. In the future, however, reporters that are currently allowed to estimate emissions using default factors from the rule might be motivated to install instrumentation to more accurately reflect their lower emissions, says Carbon Shrinks' Moore.

If that is not enough, process improvements and, potentially, carbon capture and storage (CCS) would be required. While CCS is not yet proven commercially, its implementation has fewer obstacles in the CPI than it does in power generation applications. "One of the nice things about the CPI is that the carbon dioxide is fairly pure," explains Mike Arne, assistant director at SRI Consulting (Menlo Park, Calif.; www.sriconsulting.com). "There is a tremendous amount of energy that goes into scrubbing the CO_2 from coal power plants. Compressing it and putting it into the ground requires energy, too, but not as much."

Rebekkah Marshall

Circle 16 on p. 62 or go to adlinks.che.com/29248-16 20 CHEMICAL ENGINEERING WWW.CHE.COM MARCH 2010

NewAge Industries AdvantaPure

DISPOSABLE DARLINGS

Single-use equipment and systems are a growing trend among high purity processors. Here, the experts weigh in on the pros and cons of these up-and-coming disposable components

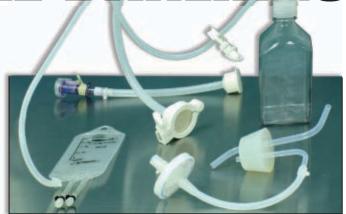


FIGURE 1. A single-use system like this one from can help reduce cleaning validation requirements, production downtime, and assembly time and costs

ot long ago, single-use bioprocessing equipment, such as disposable bags and tubing, appeared on the scene as an alternative to stainless-steel components for high-purity processing applications. Due to a fistful of benefits - including reduced costs, lower crosscontamination risk and increased flexibility — the use of disposables began to grow into a real trend among highpurity processors, especially those in the biopharmaceutical industry. With about 20% of the industry currently incorporating single-use components into at least one part of their processes, more and more processors are looking into the benefits, as well as the disposal drawbacks, and trying to decide whether single-use components and systems are right for them.

A growing trend

Laboratory and clinical use in the biotech and biopharmaceutical industries currently accounts for the largest use of disposable technology, according to Bryan Downer, solutions expert with sanitary-system-design firm CSI (Springfield, Mo.). "Use in these biotech applications makes a lot of sense because there are a lot of change outs, and disposables allow this to be done quickly and efficiently," notes Downer. Also, he explains, there's not a lot of cost involved in infrastructure changes, and a lot of equipment is available for that scale. However, according to a recent survey published by *BioProcess International* and available in detail on the Bio-Process Systems Alliance (BPSA) website (www.bpsalliance.org), adoption of single-use technologies has been significantly increasing in many processes beyond this arena.

The survey asked respondents in what processes they have adopted single-use technology and compared the data between 2008 and 2009. A marked increase can be noted in many segments. For example, use of disposables in upstream processing and media preparation jumped from 56.6% in 2008 to 62.9% in 2009. Use in cell culture and fermentation grew by leaps and bounds from 55.1 to 73.3% and in cell harvest and clarification from 43.4 to 56.2%, as well as in buffer preparation and hold from 52 to 63.8%.

Not surprisingly, implementation of all types of disposable technology has also seen growth, with even mature technologies enjoying significant increases in use. According to the survey, which asked respondents which singleuse products or technologies have been implemented and again compared figures for 2008 to 2009, bioreactors have seen the most growth, up from 31.8 to 55.9% in just one year. Bags and bioprocess containers have also seen a significant increase from 76.3 to 87.3%, followed closely by mixers, which grew from 24.2 to 34.3%, and the connectors-, pipes- and tubing category, which

rose from 56.1 to 64.7%. And, sensors, a relative newcomer to single-use technology, saw about a 7% increase in the year from 16.2 to 23.5%.

When asked why they implemented single-use technology, survey respondents cited cost savings, convenience, elimination of cleaning and sterilization cycles, reduction of contamination risk and flexibility. And with over 70% of the respondents reporting savings due to these benefits, it's easy to see why the use of disposables is growing so quickly.

Big benefits

As the biopharm industry moves away from blockbuster drugs and into the realm of "niche busters," the inherent flexibility offered by single-use technologies will likely enable success and encourage growth of the industry, says Mani Krishnan, director of Mobius Single-Use Processing Systems with Millipore (Billerica, Mass.). "The new drugs aren't going to be like the large blockbusters of the past in the sense that the molecules are going to be more specific to a smaller population," he says. He explains that this trend is occurring because, as diagnostics improve, drug developers are finding that current drugs are only effective in a fraction of patients. So the newer drugs will cater to a smaller patient population, but there are likely to be more variants of the drug that will work for the rest of the patients. "A

smaller group of patients means we're talking about going from developing drugs in a 10,000-L bioreactor to a 500 or 1,000-L unit. In addition to smaller batches, there will be changes in the way drugs will be processed," he says. "In the future, biopharm facilities will have to be more nimble so they can move from one drug product to another very fast. This type of batch processing is where we will see the adoption of single use going up significantly."

Bill Hartzell, business development manager with resin producer, Arkema (Philadelphia, Pa.), agrees. "There are huge benefits for single-use technologies as biopharm moves toward batch processing. Single use eliminates the need to clean the stainless-steel equipment between batches because you are getting systems that are all pre-sterilized," he says. "Also, you can have multiple products being made in the same facility, using the same infrastructure. And there are benefits during drug development, as well. As processors go through the phases of drug development and scale up to larger sizes, they do not have to build new infrastructure because single-use technology allows them to do multiple products under the same roof."

Another significant attraction to disposables is a reduced risk of cross contamination, says Jeff Chase, sales and marketing manager with Sani-Sure (Moorpark, Calif.). He says in a stainless-steel-based facility, contamination can be a factor, occasionally leading to loss of product. "We are told by customers in stainless-steel facilities that they lose between 5 and 9% of their product over the course of a year, but when they go to single use, that 5-9% is reduced to 1-2%," says Chase. "And when you're talking about a million dollar drug, a few percent drop in lost product is very significant."

The reason for reduced contamination risk is simple, says Ken Baker, CEO of NewAge Industries Advanta-Pure (Southampton, Pa.). If you are making one product in a stainless-steel vessel that is not dedicated and switch to another product, it can lead to cross contamination of the first product into the second. Further, it is also possible not to rinse the cleaning agents properly. "Theoretically, the cleaning proHilipore FIGURE 2. The Mobius FlexReady System family from Millipore includes equipment for buffer-media preparation, virus filtration and clarification. The company created entire systems to meet the needs of high-purity processors who are looking to move beyond single-use bags and tubing and into the realm of disposable process units

DISPOSAL OF SINGLE-USE SYSTEMS

The Disposals Subcommittee of the Bio-Process Systems Alliance recently published the Guide to Disposal of Single Use Bioprocess Systems to address the hot topic of disposal. The paper highlights the advantages and disadvantage of various disposal options, including the following:

- Landfill (treated and untreated): Landfill options offer the lowest operating costs, but are often perceived as environmentally unfriendly
- Grind, autoclave and landfilled: This practice is generally accepted as safe and helps reduce landfill volume. However, there is a significant capital cost and it requires additional handling
- Recycling: While this is environmentally appealing, it is impractical for mixed materials. Most disposables are made of mixed materials
- Incineration: This practice is also generally accepted as safe, but it may be legally restricted and can be costly
- Cogeneration: The most environmentally benign option, it offers some return on investment, but it may be legally restricted and presents the highest capital cost.
- Pyrolysis: This practice produces usable pure diesel fuel, which burns cleaner than that produced in a petroleum refinery. However, this is a very new technology, so few options are available. And its efficiency is rated as "subpar"

The paper, which discusses each option in detail is available for viewing on the organization's website.

tocol in a stainless facility should be validated for proper rinse, but maybe one batch out of 1,000 could be cross contaminated and if that batch was worth \$1 million, well, then you've just lost a million bucks," he says.

Reduced costs stemming from faster cleaning cycles and batch turnarounds are another advantage of disposable technologies. "The industry is starting to realize that single use has great benefits when it comes to reducing cleaning cycles," says Maik Jornitz, group vice president of marketing and product management for filtration and fermentation technologies with Sartorius-Stedim (Bohemia, N.Y.). "Many of the drugs are so highly potent that it requires a large volume of highly acidic cleaners to remove the residual drug components from stainless-steel surfaces and then you have to get rid of those cleaning agents," he explains. "On average it requires 8–12 h to clean and sterilize a typical 100-L tank. But if you use a 100-L disposable bag, you just rip the package open and have it set up and ready to go in 10, 20 or 30 minutes because it's pre-sterilized."

Baker reminds, "While you're spending all that time cleaning stainless steel, you're not making another batch. Turnaround is much quicker with disposables than with stainless steel, so processors can make more batches over the same period of time."

The environmental aspect

There's much deliberation in the industry with regard to the environmental impact of disposable technologies and industry associations are working hard to determine the best path to take when disposing of the products. However, most maintain that despite the amount of plastic that needs to be discarded, disposable technologies are still more environmentally sound than traditional ones.

"What you see with single-use technology is a lot more visible waste," notes Krishnan. "However, traditional stainless-steel-based facilities generate a lot of waste that you don't see because it goes down the drain."

He and other industry experts say that when comparing the carbon footprint of stainless steel and single-use processes, single-use facilities are



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200.0

150.0

100.0

50.0-0.0-

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either equivalent or carbon positive when compared with traditional facilities. Facilities that use disposables are thought to be greener due to water and energy reductions that result from skipping the typical stainlesssteel-related cleaning cycle.

As a matter of fact, SaniSure's Chase says that single-use facilities will typically see an 85 to 90% water use reduction over stainless-steel facilities. And because the water is not being heated for clean-in-place and sterilization procedures, says Chase, about 30% less energy is used to run a disposable-based plant.

Impressive sounding as this may be, most industry experts suggest that each facility do its own analysis to



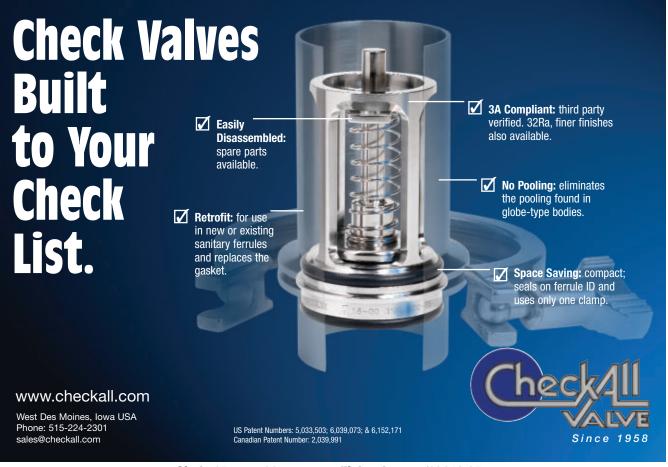
FIGURE 3. Singleuse pre-fabricated bioreactor assembly kits help make setup of single-use bioreactors even faster, which will further the growth of that segment of the market

determine if it will, in fact, find these same favorable statistics. "We encourage customers to do their own analysis because the situation can be very different in terms of what a facility pays for electricity and water use," notes Krishanan. And on top of that, there's the aspect of dealing with the "visible" waste generated by the use of disposables. How this waste is handled ranges from facility to facility and from region to region. According to the BioProcess International survey the most common form of disposal is incineration, followed by landfill, wasteto-energy and, finally, conversion for alternative purposes. And many users combine disposal methods.

"Really, the focus should not be on

what to do with the waste, but instead ought to be in understanding that there is waste in both types of processes," says Krishnan. "The question becomes how do you minimize waste? And there are easy ways to minimize the amount of plastic if you are smart about how you design your single-use systems. Most single-use systems in use today are not designed with the same amount of thought that goes into the design of stainless-steel systems. Optimal and intelligent design can significantly optimize the utilization of single-use technologies, and should be the focus, rather than what to do with all the waste that's sitting in the garbage can."

Joy LePree



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CHEMICAL CHEMICAL FACTS AT YOUR FINGERTIPS

Steam Tracer Lines

Department Editor: Scott Jenkins

Steam tracer lines are designed to maintain the fluid in a primary pipe at a designated uniform temperature. In most cases, these tracer lines are used outdoors, which makes ambient weather conditions a critical consideration.

The primary purpose of steam traps on tracer lines is to retain the steam until its latent heat is fully utilized, and then discharge the condensate and non-condensable gases. As is true with any piece of heat transfer equipment, each tracer line should have its own trap. Even though multiple tracer lines may be installed on the same primary fluid line, unit trapping is required to prevent short-circuiting.

In selecting and sizing steam traps, it is important to consider their compatibility with the objectives of the system, as traps must accomplish the following:

- 1. Conserve energy by operating reliably over a long time period
- 2. Provide abrupt periodic discharge in order to purge the
- condensate and air from the line
- 3. Operate under light load conditions

 Resist damage from freezing if the steam is shut off The cost of steam makes wasteful tracer lines an exorbitant overhead that no industry can afford.

Trap selection for steam tracer lines

The condensate load to be handled on a steam tracer line can be determined from the heat loss from the product pipe by using this formula:

 $Q = \frac{L \times U \times T \times E}{S \times H}$

Where:

- Q = Condensate load in lb/h
- L = Length of product pipe between tracer line traps in ft
- U = Heat transfer factor in Btu/ft²/°F/h
- ΔT = Temperature differential in °F
- E = One minus the efficiency of insulation (example: 75% efficient insulation or 1 - 0.75 = 0.25 or E = 0.25)
- S = Linear feet of pipe line per ft² of surface
- H = Latent heat of steam in Btu/lb

EXAMPLE

Three tracer lines at 100 psig steam pressure are used on a 20-in.-dia., 100-ft-long insulated pipe to maintain a process temperature of 190°F with an outdoor design temperature of –10°F. Assume further that the pipe insulation is 75% efficient. What is the condensate load?

Using the formula:

 $\frac{100 \text{ ft} \times 2.44 \text{ Btu/ft}^2/^{\circ}\text{F/h} \times 200^{\circ}\text{F} \times 0.25}{0.191 \text{ ft}_{\text{lin}}/\text{ft}^2 \times 880 \text{ Btu/lb}} = 72 \text{ lb/h}$

Divide by three in order to get the load per tracer line: 24 lb/h. On most tracer line applications, the flow to the steam trap is surprisingly low; therefore, the smallest trap is normally adequate. Based on its ability to conserve energy by operating reliably over a long period of time, handle light loads, resist freezing and purge the system, an inverted bucket trap is recommended for tracer line service.

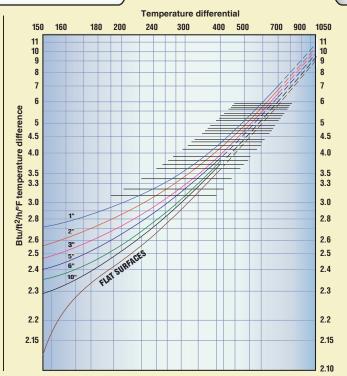


FIGURE 2. (above) The graph depicts heat loss of an uninsulated pipe. The temperature differential is derived from the process temperature minus ambient design (T=75°F)

Safety factor

Use a 2:1 safety factor whether exposure to ambient weather conditions is involved or not. Do not oversize steam traps or tracer lines. Select a steam trap to conserve energy and to avoid plugging with dirt, scale and metallic oxides.

Installation

Install distribution or supply lines at a height above the product lines requiring steam tracing. For the efficient drainage of condensate and purging of non-condensables, pitch tracer lines for gravity drainage and trap all low spots. This will also help avoid tracer line freezing (see Figure 1).

To conserve energy, return condensate to the boiler. Freeze-protection drains on trap discharge headers are suggested where freezing conditions prevail.

Material for this month's "Facts at Your Fingertips" column was supplied by Armstrong International, Three Rivers, Mich.

FIGURE 1. A con-

shows multiple tracer

lines and steam traps

Acknowledgements

densate manifold



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he 2010 Interphex Conference and Exhibition will be held April 20-22 in the Jacob Javits Convention Center in New York. Billed as North America's largest event for the biopharmaceutical manufacturing industry, the show is sponsored by the International Society for Pharmaceutical Engineering (ISPE). Its content is organized into four tracks - pharmaceuticals, biotechnology, generic drugs and contract services - and will offer various educational seminars. The conference will feature a keynote address by news anchor Chris Matthews, host of "Hardball," which airs on MSNBC. Attendees are encouraged to visit Chemical Engineering staff at booth T1800. The following items are among those to be displayed in the event's exhibit hall:

This valve and sensor line is designed for hygienic processes

The Element line of valves, sensors and controllers (photo) is designed to combine the properties of engineered polymers with those of stainless steel. The equipment has no coatings, pockets or external pneumatic lines to adversely affect cleanliness. The valves include bright visual feedback via signal LEDs or a backlit graphic display. The line can handle steam, corrosives and abrasive fluids. Booth 2537 — Burkert Fluid Control Systems, Charlotte, N.C. www.burkert-usa.com

An ink-jet printer that is designed for heavy-duty applications

This company's 1610 small-character continuous ink-jet printer (photo) is designed for high-speed, large-output applications, and can print up to five lines of high-resolution print at speeds up to 960 ft/min. The 1610 model features a vapor recovery system that reduces solvent consumption and allows longer runs between fluid changes. Also, the device has a clean-flow printhead design that reduces ink buildup. Key ink-system components are designed to be replaced by customers in 30 min without the need for a visit by a trained technician. Booth 2310 - Videojet Technologies Inc., Wood Dale, Ill. www.videojet.com



Videojet Technologies

Replace solenoid valves without

changing wiring or signal levels

The new Type 3738 self-diagnostic.

electronic valve position monitor

(photo) can be used for on/off valves in

all applications. It indicates the end po-

sitions and controls the actuators. Be-

cause of its contactless, magnetoresis-

tive sensor system and an integrated

microprocessor, the valve position

monitor can be configured at the push

of a button. Said to be the first device

of its kind, the monitor works for sig-

nals that meet the Namur standard

and is powered by a two-wire supply.

As a result, the valve position monitor





can replace solenoid valves and limit switches without having to change the wiring or signal levels. At the same time, it includes self-tuning and diagnostic functions. Booth 3042 — Samson AG, Frankfurt am Main, Germany www.samson.de

Use this pressure gage for alternate units of measure

This series of digital sanitary pressure gages (photo) offers an alternative to conventional mechanical pressure gages, allowing users the possibility of obtaining data with alternate units of measure. The instrument can also generate a dampened display value and a minimum or maximum value. Gages can be equipped with switches or a 4–20-mA output, allowing the gage to do the work of a pressure switch or transmitter, while providing a local reading on a large display. It features a stainless-steel, weatherproof housing. Booth 1551 — Ashcroft Inc., Stratford, Conn.

www.ashcroft.com

OCS Checkweighers

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This weigher improves simplicity and user ergonomics

The HC Avantgarde checkweigher series (photo) features a modern ergonomic design for more userfriendly operation. Based on technology from earlier models, the series is intended for pharmaceutical production. A stainless-steel control panel rotates and tilts for simpler use, and an open "pyramid design" offers easy cleaning. Booth 755 — OCS Checkweighers Inc., Snellville, Ga. www.ocs-cw.com

Complete 400 plunger-rod assemblies per minute with this machine

Type 607 high-speed plunger-rod insertion machines boast an output of 400 assemblies per minute, and are combined with a label dispenser. The Type 607 is a continuous rotary-type machine with servo-controlled motion of the main drive and servo-controlled syringe rotation. The servo of the rotation moni-



tors the torque precisely, thus avoiding motion by the plunger stopper. The mechanically controlled, plunger-rod insertion offers repeatability. Additional features include a fully integrated vision system for verification of component assembly and a touchscreen user interface. Booth 2828 — Bausch Advanced Technologies Inc., Clinton, Conn. www.bausch-group.com

Lower contamination risk with this vacuum

The CFM 3306 pharmaceutical vacuum cleaner (photo) is designed for fixed installations with drug-processing equipment, including milling, filling, dedusting, polishing and packaging machines. It collects ultrafine powders and debris straight from the source, which reduces contamination risk. The 3306 is ideal for cleanrooms

down to Class 100. Booth 1565 — Nilfisk CFM, Malvern, Pa.

www.nilfisk-advance.com

Capsule filling in continuous or intermittent mode

The Labby is a modular, compact capsule filler that is fully automatic and capable of operating in either continuous or intermittent mode. The machine can fill up to 3,500 capsules per hour and



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Busch Liquid Ring Vacuum Solutions

for process applications in the chemical industry

handles many powder types. The unit features a micro-weighing control system that maintains system accuracy to within 0.1 mg of the net product weight. The Labby has a small footprint that makes it ideal for small batch outputs, clinical trials and R&D. Booth 1519 — *MG America Inc., Fairfield, N.J.* **www.mgamerica.com**

An inline viscometer based on fluid pressure

This company's new viscometer (photo, p. 24D-4) is based on a new measuring principle — dynamic fluid pressure — which is proportional to viscosity. The inline viscometer is designed to be immersed in the production liquid directly within the pipework or tank. Pressure drops and flowrate decreases do not affect results. The instrument can accurately determine fluid viscosities from 1 to 10,000 cP. Booth 2761 — Anton Paar USA, Ashland, Va. www.anton-paar.com

Protect against counterfeits with this system

This company offers a low-cost, Internet-based system for identifying counterfeited pharmaceutical products using standard flatbed scanners or mobile phone cameras. The system captures an image of a pharmaceutical product and sends it to a server that performs pattern-matching and returns a "genuine or fake" verdict. The system is appropriate for various layers of product packaging, including carton boxes, aluminum blister packs or the tablets themselves. Booth 725 — AlpVision SA, Vevey, Switzerland **www.alpvision.com**

This valve is designed with an easy user interface

This inline radial diaphragm valve is a wireless system with an easy user interface for applications in the pharmaceutical and biotechnology industries. It installs rapidly, assembles without tools and can be oriented anywhere within 180 deg of rotation. The valve is available with manual or pneumatic actuation, and does not need adjusting or re-tightening. Booth 2001 — Asepco Advanced Aseptic Processing, Mountain View, Calif. www.asepco.com



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This company's services take a proactive approach

VigilantPlant services revolve around this company's plant automation platforms. VigilantPlant aims to enable an ongoing state of operational excellence in which informed and attentive plant personnel optimize plant and business performance. The service concept comprises three service components: Opportunity Identification; Solution Implementation; and Lifecycle Effectiveness. Booth 2844 — Yokogawa Corp. of America, Newnan, Ga.

www.yokogawa.com

Produce small batches with this system

A modular system from this company is designed for smaller batches of pharmaceuticals, as well as for process development and clinical studies. The table-top machine has a modular design that allows for reproducible filling and closing of syringes, vials, cartridges, eyedrop and nasal-spray bottles. Possible filling systems include rotary piston pumps, disposables, peristaltic pumps and more. Booth 665 -Groninger USA LLC, Charlotte, N.C. www.groningerusa.com

This pallet positioner has a self-leveling design

The EZ Loader automatic pallet positioner has a self-leveling design that keeps pallet loads at a convenient working height automatically. An integral rotating platform allows nearside loading to eliminate reaching. The EZ Loader utilizes captive air operation, obviating the need for external hydraulics. The product is especially suited to tough environments in the pharmaceutical and food manufacturing and packaging industries. Booth 1068 — Bishamon Industries Corp., Ontario, Canada

www.bishamon.com

Prevent the spread of fire with this pinch valve

A newly introduced, pneumatically operated, explosion-isolation pinch valve (EIPV) from this company offers a means to prevent deflagrations from



propagating through interconnecting pipes or conveying lines. This explosion-isolation method helps protect downstream process equipment and nearby operating locations from accidents. The EIPV consists of a heavyduty, cast-valve body with a rugged elastomeric sleeve. In tests, the EIPV blocked explosion pressures of 3 bar, says the company. It can be mounted either vertically or horizontally and is virtually maintenance-free. Booth 1360 — Fike Corp., Blue Springs, Mo. www.fike.com

Expanded functionality is a feature of this manufacturing software

TrakSYS performance management software allows users to manage and benchmark their manufacturing operation from material planning to finishedproduct tracking. A new version has more user-definable and configurable analytics and vendor-independent application connectivity. Booth 2841 -Parsec Automation Corp., Brea, Calif. www.parsec-corp.com

This steam-trap valve is designed for high-purity processing

Designed exclusively for high-purity processing, the Opus steam-trap valve is a multifunctional valve that provides three states of fluid control, and has an integrated design that eliminates more complex piping schemes. Opus valves are available in a wide selection of sizes, with several different trap connections and in four materials. Booth 2867 - SVF Flow Controls Inc., Santa Fe Springs, Calif. www.svf.net

Scott Jenkins

Anton Paar USA

Thermo Fisher Scientific

MARCH New Products

MR9600

ABB Analytical

A benchtop FTIR for near-infrared analysis

Launched last month, the MB-600-PH FT-NIR (Fourier transform, near-infrared) spectrometer (photo) is a benchtop analyzer suitable for a broad range of applications, including quality control analysis, raw material identification and qualification, R&D and inline process analytics. The instrument combines the attributes of a research-grade analyzer — in particular an outstanding signal-to-noise ratio - while maintaining a minimal cost of ownership, says the manufacturer. Its aluminum casting also provides a level of protection needed for intensive use in an industrial environment. — ABB Analytical, Québec, Canada www.abb.com/analytical

A flowmeter that ensures reproducible chromatography

The GFM Pro Flowmeter (photo) continuously measures gas flows in realtime for accurate and reproducible chromatography analysis. With the capacity to measure both positive and negative vacuum flows from ± 0.5 to 500 mL/min, the CE-certified probe can measure volumetric flow of any gas with an accuracy of $\pm 2\%$. The unit also protects itself against excessively high flowrates by deploying an automatic shutoff when the measured flow exceeds 600 mL/min. — *Thermo Fisher Scientific Inc., Milford, Mass.* www.thermofisher.com

Emerson Process Management

GFM PRO

ermo

Abrasive slurries are not a problem for this disc valve Designed for heavy scaling, abrasive

Larox Flowsys Oy

Designed for heavy scaling, abrasive and corrosive slurries, the LDR Rotory Disc Valve (photo) is suitable for applications in mineral processing, power generation, and the graveland-sand, pulp-and-paper, chemical and mining-and-metal industries. The patented design allows the valve to cycle in heavy-scaling slurries without sticking or leaking and with minimum wear. It is available with pressure ratings up to 100 bar. — Larox Flowsys Oy, Lappeenranta, Finland www.larox.fi/flowsys

A machinery protection solution that's fast to setup

Machinery protection and prediction of critical mechanical equipment has been integrated into the DeltaV digital automation system (photo). This new capability directly supports users' goals for improved availability and performance. This integrated machinery protection-and-prediction solution easily connects to the DeltaV system in three simple steps that take less than 10 min. Asset parameters are scanned, selected and imported into DeltaV from AMS Suite predictivemaintenance software and the CIS 6500 Machinery Health Monitor. After import, the DeltaV alarm banner is automatically populated and the system is fully configured with function blocks that can be further used in control strategies. — *Emerson Process Management, Baar, Switzerland* **www.emersonprocess.eu**

Test wet or dry powders with this sieve-tower analyzer

The Laboratory/Pilot Sieve Tower (photo. p. 24I-2) tests both wet and dry material, analyzing a wide range of product samples, including ceramics, foodstuffs, pharmaceuticals, and metal and paint powders. Dual drive controls assure effective "throughout" for up to eight full-frame sieves or 16 half-frame

New Products

sieves with mesh sizes as small as 20 microns (635 mesh). — Cleveland Vibrator Co., Cleveland, Ohio www.clevelandvibrator.com

Keep instrumentation free of condensation with this heater

This new range of steam heaters (photo) for field-instrumentation-protection applications offers a choice of vertical, horizontal or panel mounting styles. With four outputs ranging from around 65 to 490 W, users can select the appropriate heater for all common enclosure-scale freeze and condensation protection or temperature-maintenance applications. The steam elements are identical in size to those of the firm's electrical heating range, but come with a press-fitted, 12-mm OD stainless-steel tube for connection to the steam or hot-water service line. ---Intertec Instrumentation Ltd., Sarnia, Ont., Canada

www.intertec-inst.com

Track bio-aerosols with this validated device

The new generation of SAS Isolator, stainless-steel air sampler simplifies bio-aerosol monitoring in isolators and other controlled environments. The system is composed of two parts: an aspiration chamber that is positioned within the isolator or near production lines; and an external command unit. The system is validated according to ISO 14698, and the stainless-steel construction is suitable for sterilization by vaporized hydrogen peroxide and peracetic acid. — International PBI S.p.A., Milan, Italy

www.internationalpbi.it

Shred and granulate plastics and wood with one machine

Now it is possible, in one single sizereduction step, to both shred and obtain small granule sizes with the HB Series granulator. The design of this machine combines a feed hopper and a hydraulic ram with a granulator. The special design of the grinding chamber and the high cutting frequency enables the machine to transform bales, cut-open film rolls, mingled packs and extremely large and thick-walled purgings into the finished product in one step. Four versions are available,



Intertec Instrumentation

Michael Smith Engineers

with throughputs of 0.5–3.0 ton/h to 1.2–6.0 ton/h. — Herbold Meckesheim GmbH, Meckesheim, Germany www.herbold.com

Larger members for this family of small pumps

The established range of HNP positive displacement, internal-gear pumps has been expanded to include new, larger pumps and more drive options. The 11500 Series (photo) deliver output flows of 0.2-1,150 mL/min and differential pressures between 0-60 or 0-150 bar, depending on the viscosity of the process fluid. Three motor options are available: a.c., d.c. and an ATEX-rated, explosion-proof a.c. version for hazardous areas. — *Michael-Smith Engineers Ltd., Woking, U.K.* www.michael-smith-engineers.co.uk

Hearing protection with hybrid design

The new Pilot push-in earplug features a hybrid design that combines the performance and cost savings of

multiple-use earplugs with the superior comfort of single-use earplugs. Pilot inserts easily into the ear with a simple fingertip twist of the nonobtrusive Navigation Stem. Its soft, pearl-skinned polyurethane foam construction is resilient and easy to clean. With NRR 26 rating, Pilot provides protection in medium-to-low noise environments (95 dB or less). — Howard Leight/Sperian Hearing Protection, LLC, Smithfield, R.I.

www.howardleight.com

Keep explosions from propagating with this pinch valve

The Explosion Isolation Pinch Valve (EIPV) is an economical, yet reliable form of explosion isolation — a method for preventing deflagration propagation through interconnecting pipes or conveyor lines. The EIPV consists of a cast valve body containing an elastomeric sleeve. Upon explosion detection, compressed air is released at high speed and within milliseconds, the sleeve is pinched to full closure,



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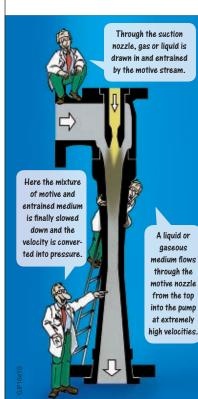
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Jet pumps form the core of jet vacuum pumps, steam jet cooling plants, degassing systems, condensation systems and heating/cooling units. They can be used in many different processes for conveying, recompressing and mixing of gases, vapours and liquids and for evacuating of tanks by drawing off gases, vapours

and solvents.



GEA Process Engineering

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New Products

stopping explosion propagation beyond the valve. The valve has been proven to block explosion pressures of 3 bar. - Fike, Blue Springs, Mo. www.fike.com

A new globe valve comes in a range of sizes

The new 537 manually operated globe valve (photo) uses the same valve body as the company's 534 and 532 actuator designs. Valve bodies in nominal sizes DN 15-50 in cast-stainless-steel 1.4408 and SG iron GGG 40.3 (EN-GJS-400-18-LT) are available. The maximum operating pressure is 25 bar for DN 15-40 sizes, and 16 bar for DN 50. Standard versions can handle temperatures up to 180°C, and the PTFE gland packing is suitable for steam. — GEMÜ Gebr. Müller Apparatebau GmbH & Co. KG, Ingelfingen-Criesbach, Germanv

www.gemue.de

Mix solids as if they were fluids with this blender

The Vibra Blender (photo) uses a unique combination of a rotating paddle and material fluidization to blend dry solids with the ease of liquids, according to the manufacturer. Gentle, controlled vibration of the mixing trough fluidizes ingredients on entry where a cut-and-fold mixing rotor provides agitation. Materials flow through the Vibra Blender by gravity making it completely self-regulating to changes of infeed rates. Units are available in capacities up to 1,500 ft³/h. — Vibra Screw. Totowa. N.J. www.vibrascrew.com

Advanced cartridge filters for collecting dust and fumes

The Cam-Airo filter cartridge is engineered to solve nearly all dry-dustand fume-collection challenges. Unlike horizontal designs that sacrifice nearly 30% of the filter media area over time, Cam-Airo's vertical design optimizes virtually 100% of the filtermedia area. The cartridge collector is fabricated in welded sub-assemblies,



Vibra Screw



GEMÜ

flanged and externally reinforced, thereby avoiding horizontal ledges that can obstruct airflow, retain dust and cause bridging. Cartridges are available in a variety of materials and surface treatments, with filtration areas of 325 or 245 ft² per cartridge. - Camcorp Inc., Lenexa, Kan.

www.camcorpinc.com

These burner valves now have TÜV approval

The Jamesbury series 7000/9000 flanged ball valve are automated shutoff valves designed not only to meet EN161 and EN264, as approved by TÜV Rheinland, but also provide protection against fire and explosive hazards during the operation of gasand oil-burning equipment. When the electrical signal is interrupted, or when there is a loss of air pressure, these burner valves close within 1 s to isolate the gas or oil flow. This action may be initiated either by safety trip or normal shutdown sequencing. The units are also suitable for use in safety loops with SIL compliance according to ISO 61508. - Metso Automation Inc., Helsinki, Finland

www.metso.com/automation Gerald Ondrey

People

WHO'S WHO



Vanhove

Andre Vanhove is promoted to global HPI product applications leader for **GE Water** (Herentals, Belgium).

Lawrence Sloan becomes president and CEO of the Society of Chemical Manufacturers and Affiliates (SOCMA; Washington, D.C.).

Haws Corp. (Sparks, Nev.) promotes *Sallie Haws* to chairman of the board and *Thomas White* to president.

Jacob Söhne GmbH (Porta Westalica, Germany) names *Uwe*



Corbin

Braun technical director and *Ralf Borcherding* commercial director with responsibility for global sales.

NanoGram Corp. (Milpitas, Calif.) names *Dave Corbin* CEO.

Dave Wareheim becomes bioprocess subject matter expert at **Integrated Project Services** (Lafayette Hill, Pa.), a full-service engineering, construction and commissioning firm.

Continential Disk Corp. (Liberty, Mo.) and subsidiary **Groth Corp.**





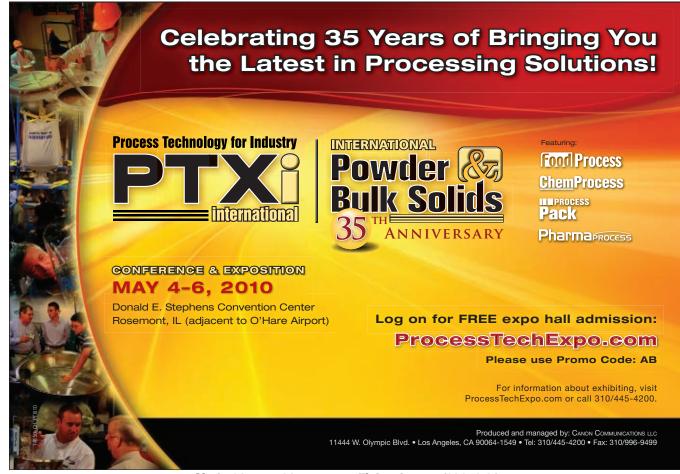
Wareheim

(Stafford, Tex.) name *Steven Breid* vice-president, sales and marketing. Groth also appoints *Mitchell Anderson* director of operations.

Luis Miguel Salinas becomes vicepresident, principal in charge of the environmental department for engineering-design firm **Paulus**, **Sokolowski & Sartor** (Warren, N.J.).

Jason Rainbird is named general manager of **RO UltraTec (Europe)** Ltd. (Kent, U.K.).

Suzanne Shelley



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Cover Story

Saving Energy In Regenerative Oxidizers

Catalysts can reduce use of auxiliary fuel in regenerative oxidizers

Grigori A. Bunimovich and **Yurii Sh. Matros** Matros Technologies

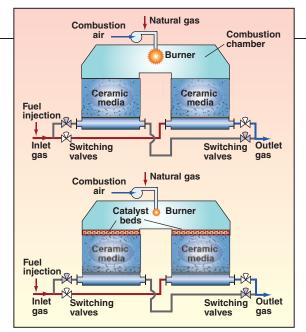


FIGURE 1. Regenerative thermal oxidizers (top) and regenerative catalytic oxidizers (bottom) have similar designs

xidation of volatile organic compounds (VOCs) in regenerative thermal oxidizers (RTOs) is the most common process in the chemical process industries (CPI) for removing diluted emissions of VOCs and carbon monoxide from stationary airpollution sources. Thousands of RTOs operate in automobile, engineered wood, chemical and other industries.

RTOs combine gas-phase thermal oxidation of VOCs with regenerative heat exchange. Heat energy of exhaust gas is efficiently reused for heating the inlet gas. Typical process temperatures are between 1,400 and 1,800 °F. Despite the high degree of energy recuperation, many large RTOs require substantial consumption of auxiliary fuel.

An alternative to thermal oxidation is catalytic oxidation of VOCs, which runs at much lower temperatures — about 500-900 °F. A regenerative catalytic oxidizer (RCO) uses the same operating principle as RTO, but consumes substantially less auxiliary fuel. The authors have found that in many situations, RTOs can be retrofitted to RCOs. Energy savings after such a retrofit can quickly justify the costs for the catalysts and installation.

This article discusses basic RTO and RCO technology with an emphasis on thermal efficiency and energy balance. A method for quickly estimating auxiliary fuel consumption is suggested. The requirements of VOC oxidation catalysts are reviewed, along with the issues of catalyst performance maintenance, deactivation and regeneration. The retrofit of RTO to RCO is exemplified for various industries.

Design and operation

The simplest RTO arrangement (Figure 1, top) comprises a horizontal combustion chamber connecting two vertical heat-exchange canisters loaded with refractory material, such as ceramic Intalox saddle- or honeycombmonolith blocks. A burner installed in the middle of the combustion chamber provides heat for the oxidizer startup. Fuel combustion products and process gas are well mixed within the combustion chamber. The bed downstream from the chamber collects a fraction of the heat energy through heat exchange with the process gas. Fast-actuating valves reverse flow direction in the system every 1-3 min. The bed that collects heat during the previous cycle returns it to the process gas entering the oxidizer. Another bed collects the heat from the outlet gas. Because of periodic flow reversals, the combustion energy is effectively trapped in the beds and the system temperature gradually rises. The burner firing is reduced upon reaching the required temperature.

According to temperature profiles calculated for an RTO (Figure 2, left), the periodic flow reversal results in a nearly linear temperature distribution along the length of gas passage in packed beds. The temperature increases in the inlet bed and symmetrically decreases in the outlet. Stepwise temperature rise in the middle of the combustion chamber reflects energy addition due to burner firing. Temperature fluctuations during the cycle are small because of the ceramic media's high heat capacity. Conversion of VOCs occurs mostly in the combustion chamber, where the destruction and removal efficiency (DRE) increases and achieves completion.

The system can operate fuel-free if the energy released during the exothermic VOC oxidation exceeds overall heat removal with exhaust gas and heat losses to the environment. When the concentration of VOCs is low, the system requires energy addition.

Most existing RTOs built from the 1980s through the 2000s are burnerfired. That is, they use continuously fired startup burners for maintaining combustion of low concentrations of VOCs. The burner operates as an autonomous combustion unit, consuming oxygen from ambient air that is pushed by a separate fan. The energy for heating the combustion air and fuel mix in the combustion chamber is not recuperated, which contributes to overall fuel consumption.

Many recently built RTOs are fuel-injected, which means they operate with direct injection of natural gas into the inlet duct. The natural gas is oxidized together with VOCs, consuming oxygen in the process stream without separate feeding of combustion air. The burner is still required for oxidizer startup because fuel injection is activated upon achieving the temperature necessary for the combustion of natural gas. The process typically requires higher oper-

NOMENCLATURE

Delta T definitions:

$$\Delta T_{VOC} = \frac{\sum_{i} M_{VIOCi} q_{VOCi} DE_{i}}{F_{PG} C_{PG}} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{BC} = \frac{q_{fuel}}{C_{CM} \left(1 + k_{SC} k_{AE}\right)} \qquad \Delta T_{HL} = \frac{k_{HL} A}{F_{PG} C_{PG}} \qquad \Delta T_{HL} = \frac{k_{HL}$$

- a Specific surface area of heat exchange material, ft²/ft³
- Oxidizer external surface area, ft² Α
- Specific heat capacity, Btu/(lb°F) С
- $C = c \rho_0$ Specific heat capacity per unit of standard gas volume, Btu/(ft^{3°}F)
- DE; Destruction and removal efficiency for *i*-th VOC
- F Gas flowrate at standard conditions, scf/h
- **k**_{AE} Air excess ratio
- Coefficient of heat transfer, Btu/ft²h k_H
- Heat loss factor, Btu/ft²h **k**_{HL}
- **k**_{HT} Ratio between number of thermal units for catalyst and ceramic beds

- ksc Molar air/fuel ratio for stoichiometric combustion Depth of bed, ft
- MVOCi Flowrate (loading) for VOC indexed *i*, lb/h
- Lower heating value for combus**q**fuel tion of one standard cubic foot of gaseous fuel, Btu/ft³ qvoci Lower heating value for
- oxidation of i-th VOC, Btu/ft3 Q Component of heat balance, Btu/h
- Inlet temperature, °F T_{in}
- Maximum temperature in the T_{max} oxidizer, °F
- Tout Outlet temperature, °F



$$\eta_{RCO} = \eta \frac{1 + k_{HT}}{1 + \eta k_{HT}} \quad \text{at} \quad k_{HT} = \frac{k_{H,CAT} a_{CAT}}{k_{H,CER} a_{CER}} \frac{L_{CAT}}{L_{CER}}$$

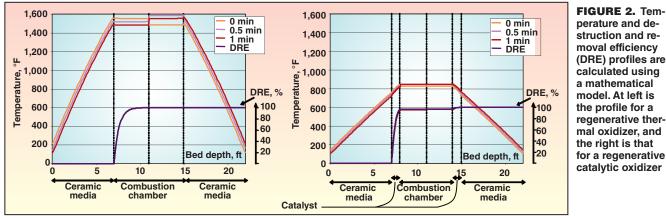
- η Thermal efficiency of fuel-injected regenerative oxidizer
- Thermal efficiency of burner-fired η**в** regenerative oxidizer
- Gas density at standard conditions, ρο lb/ft³

Subscripts:

AF BF	Auxiliary fuel Burner fired oxidizer	HL
CAT	Catalyst	
CER	Ceramics	PG

CM Combustion air/fuel directed to the burner Heat losses to the environment

Process gas



perature and destruction and removal efficiency (DRE) profiles are calculated using a mathematical model. At left is the profile for a regenerative thermal oxidizer, and the right is that for a regenerative catalytic oxidizer

ating temperatures than burner-fired RTOs, but the fuel consumption is still lower because no preheating of combustion air is required.

The RTO retrofit to RCO is straightforward [1-3]. In most applications, a bed of catalyst is placed over the bed of existing ceramic material in each RTO canister (Figure 1, bottom). If necessary, the top fraction of the ceramic media can be removed to make space for the catalyst.

The process profiles in an RCO (Figure 2, right) are similar to an RTO (Figure 2, left), except that VOC conversion occurs in catalyst beds, and at much lower temperatures (800 °F instead of 1,500 °F in this example). The outlet temperature in an RCO is lower than in an RTO, which decreases energy losses with exhaust gas.

In a simple, two-canister RTO, the overall conversion of VOCs is effectively reduced due to periodic displacement of a cold, non-reacted gas collected under the inlet bed every cycle. Many units apply various modifications of the simple design to alleviate the negative effect of VOC displacement. The most popular technique involves the addition of a so-called odd canister. The overall cycle includes canister purge prior to its operation in outlet mode. During the purge, the canister is washed by a fraction of gas taken from the oxidizer exhaust or the combustion chamber. The displaced VOCs are directed to the combustion chamber or recycled to the oxidizer inlet.

Fuel consumption

Fuel consumption in an RTO is usually linked to thermal efficiency, which is often defined as:

$$\eta = \frac{T_{\max} - T_{out}}{T_{\max} - T_{in}} \tag{1}$$

Where T_{max} is the maximum process temperature, equivalent to the temperature setpoint in a combustion chamber, and T_{in} and T_{out} are inlet and outlet temperatures of process gas. Equation (1) was transferred from the theory and practice of regenerative heat exchangers [4], where T_{max} and T_{in} are read as inlet temperatures of heat transfer

fluids during hot and cold periods, and T_{out} is the average outlet temperature during the hot period. Further analogy with heat exchangers implies that the efficiency should be independent of the other parameters, which do not affect the heat transfer in packed beds. This means, for example, that the RTO should adjust itself and achieve the same thermal efficiency at any inlet temperature, combustion-chamber setpoint and VOC concentration. At high temperatures sufficient for achieving complete combustion of VOCs and injected fuel, fuel-injected RTOs operate this way. The measure of thermal efficiency by Equation (1) can be applied for odd-chamber RTOs or RCOs typically operating at a low flowrate of purge gas. It cannot be used for burnerfired RTOs or RCOs where the outlet temperature depends significantly on the flowrate of combustion air.

Fuel injected oxidizers The heat balance for the fuel-injected oxidizer is:

$$-Q_{PG} - Q_{HL} + Q_{VOC} + Q_{AF} = 0$$

Cover Story

Where Q_{PG} and Q_{HL} are energies spent for the rising temperatures of process gas and compensating heat losses to the environment, and Q_{VOC} and Q_{AF} are energies generated by the combustion of VOCs and auxiliary fuel. At constant process-gas heat capacity, the energy terms in the heat balance given by Equation (2) can be converted to temperature differences ("delta Ts") using the following replacement:

$$\Delta T = \frac{Q}{F_{PG}C_{PG}} \tag{3}$$

where F_{PG} and C_{PG} are the process gas flowrate and average volumetric specific-heat capacity calculated between the inlet and outlet temperatures.

The temperature difference between outlet and inlet gas in the fuel-injected RTO can be expressed through the difference between maximum and inlet temperatures using Equation (1). Then Equation (2) can be rewritten as:

$$\Delta T_{AF} = \frac{Q_{AF}}{F_{PG}C_{PG}}$$

$$= (T_{max} - T_{in})(1 - \eta) + \Delta T_{HL} - \Delta T_{VOC}$$
(4)

where ΔT_{AF} and ΔT_{VOC} are the temperature rise of process gas due to combustion of auxiliary fuel and VOCs, and ΔT_{HL} is the temperature drop due to heat losses from the oxidizer walls. Formulas for calculating delta-Ts are provided in nomenclature.

Burner-fired oxidizers Compared to Equation (2), the heat balance for a burner-fired oxidizer also includes the energy spent heating the combustion air and fuel. Still, the temperature rise associated with auxiliary fuel consumption can be estimated using the following equation:

$$\Delta T_{AF,BF} = \frac{(T_{\max} - T_{in})(1 - \eta) + \Delta T_{HL} - \Delta T_{VOC}}{1 - \frac{(T_{\max} - T_{in,CM})}{\Delta T_{BC}}}$$
(5)

In Equation (5), $T_{in,CM}$ is the temperature of combustion air and fuel directed to the burner. The temperature difference ΔT_{BC} represents the quotient of auxiliary fuel consumption and combustion-mixture heat-capacity rate calculated between $T_{in,CM}$ and T_{max} . Equation (5) is obtained assuming that the energy spent for heating the combustion mixture from $T_{in,CM}$ to T_{max} is the only contribution for fuel consumption increase in burner-fired RTOs compared to fuel-injected ones.

It is important that the thermal efficiency η in Equation (5) is determined for fuel-injected oxidizers. Actual or experimental efficiency for burner-fired oxidizers (η_{BF}) calculated using

Equation (1) is less than the same for fuel-injected ones. The generic thermal efficiency (η) applied in Equation (5) can be recovered from the experimental efficiency in a burner-fired unit ($\eta_{_{RP}}$) using the following estimation:

$$\eta = \eta_{BF} \left(1 + \frac{1}{\Delta T_{BC}} \times \frac{T_{out} - T_{in} - \Delta T_{VOC} + \Delta T_{HL}}{1 - \frac{T_{out} - T_{in,CM}}{\Delta T_{BC}}}\right)$$
(6)

Equations (4) and (5) also can be used for estimating the fuel consumption in an RCO, provided that the thermal efficiency accounts for the heat transfer properties in the catalyst bed. The nomenclature includes a formula for calculating the thermal efficiency for a retrofitted, fuel-injected RTO, accounting for a ratio between depths, specific surface areas, and coefficients of heat transfer in ceramic and catalyst beds.

How fuel consumption varies Figure 3 illustrates fuel consumption in burner-fired and fuel-injected RTOs and RCOs determined from Equations (4) and (5). Temperature rise due to VOC oxidation (ΔT_{VOC}) is considered variable, while other parameters are taken as constant. ΔT_{VOC} is directly proportional to the overall VOC concentration and is substituted for this parameter in further discussion.

The example in Figure 3 comprises a typical state-of-the-art RTO with a thermal efficiency of 95%. The process gas, fuel and combustion air have the same inlet temperature: 70 °F. The temperature set-point in the combustion chamber (T_{max}) is taken to be 1,500°F for both fuel-injected and burner-fired RTOs, even though the fuel-injected RTOs typically require higher temperatures. For burner-fired RTOs, ΔT_{BC} is estimated at 4,785 °F, assuming a methane combustion at 0% excess combustion air. The unit heat

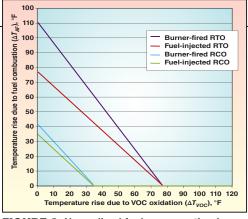


FIGURE 3. Normalized fuel consumption is plotted against VOC oxidation temperature rise

losses are assumed to be equivalent to the process gas temperature reduction ΔT_{HL} = 6 °F. The RCO is assumed to operate at a combustion-chamber temperature of 750 °F and a thermal efficiency of 95.7%. These values are common for a number of RTOs that have been converted to RCOs using commercial base-metal catalysts.

According to Figure 3, fuel consumption decreases linearly with an increase in VOC concentration. Crossing the calculated lines with the horizontal axis reflects the transition to fuel-free operations, when the necessary temperature can be supported by the exothermic reaction of VOC oxidation without auxiliary fuel consumption. The RCO provides self-sustaining operations at VOC concentrations twice as low as an RTO.

Figure 3 also shows that using the fuel-injected RTO reduces energy consumption by about 30% over the burnerfired RTO at very low (close to zero) VOC concentration. Fuel injection efficiency decreases at higher VOC concentration. From another result in Figure 3, the difference between fuel consumption in burner-fired and fuel-injected RCOs is much smaller than in RTOs.

Using the catalyst in the burnerfired RTO reduces fuel consumption by 63% at low VOC concentrations. Relative fuel savings increase with an increase in VOC concentration, and achieve 100% at VOC concentrations between the self-sustaining limits for RCOs and RTOs.

It may be too expensive to realize the catalytic process in a fuel-injected RTO because methane has a relatively low oxidation rate over commercial catalysts. A feasible RTO retrofit scenario can involve fuel injection shutoff with subsequent operation at a continuously fired start-up burner. According to Figure 3, the burner-fired RCO consumes half the fuel of a fuel-injected RTO.

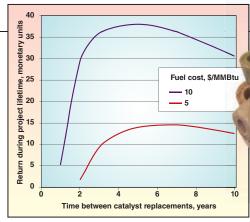


FIGURE 4. Economic impact of retrofitting: expensive fuel justifies more frequent catalyst replacement

Electricity consumption

Using a catalyst can reduce electricity consumption in addition to auxiliary fuel. RTOs typically include fans for transporting the process gas through the system. The fan power (kW_{fan}) is proportional to flowrate (F) and total system pressure drop (Δp) :

$$kW_{_{fan}} \propto F \Delta p$$

(7)

Ceramic beds in retrofitted RTOs operate at lower temperatures and have reduced pressure drops as compared to the original unit. If temperature is expressed in Fahrenheit, the approximate ratio between the ceramic bed pressure drops in RCOs and RTOs is:

$$\frac{\Delta p_{cer}^{RCO}}{\Delta p_{cer}^{RTO}} \approx \left(\frac{T_{\max}^{RCO} + T_{in} + 919}{T_{\max}^{RTO} + T_{in} + 919}\right)^n$$

where the power coefficient (n) depends on the type of ceramic media. For random packing, such as Intalox saddles, $n \approx 1$; for straight channel monolith, $n \approx 1.7$. The total pressure drop reduction can reach 15–35% depending on process temperature and type of ceramic media. This may compensate for the pressure drop increase after catalyst addition.

There is an additional component of energy savings for systems that use induced-draft fans installed in the RTO outlet — the fan flowrate (F) decreases at low outlet temperature in the RCO, which results in a corresponding decrease in electricity consumption.

Generally, the retrofit requires careful analysis of pressure drop and existing fan capacity. As a rule of thumb, the catalyst size should be comparable with the size of the ceramic media.

Catalyst general requirements

Catalyst design and selection should strike an optimal balance among activity, selectivity, lifetime, mechanical

FIGURE 5. Manganese oxide catalyst Z-2 can be used for retrofits

strength and cost. For VOC control in regenerative oxidizers, the catalyst should satisfy the following requirements:

- High activity in oxidation of specific VOCs present in the treated gas
- Long lifetime in the presence of catalytic poisons, such as alkali and alkaline-earth metals in composite-board, silicon-organics in semiconductors, sulfur in fuel-ethanol production, and metals in paint pigments for the automobile industry
- Low pressure drop, permitting the addition of catalyst without an increase in power consumption
- High thermal stability, allowing periodic burnouts of organic deposits collected in RTO beds and ducts or withstanding uncontrolled temperature increase caused by fluctuations in VOC concentration

 High mechanical strength The noble metal catalysts used in industry for decades typically comprise active metals incorporated within a thin layer of porous alumina washcoat deposited over a solid ceramic carrier. The carrier can be made as a ceramic saddle or monolith and often has the same shape and size as RTO ceramic media. Most noble metal catalysts contain platinum, which provides high resistance to poisoning by sulfur and other contaminants. Platinum is expensive, however and even a small percentage raises catalyst price. Recycling metals from used catalysts can reduce ownership costs somewhat. In small units (up to 1,000 scfm), the catalyst cost represents a relatively small portion of the total price, which makes noble metal catalysts beneficial due to their low bed volume and availability in monolithic form. However, such catalysts may be less competitive as flowrate increases. In large capacity oxidizers, the catalyst contributes a larger portion to the total cost of the system, so economics favors less expensive base-metal catalysts.

The base-metal catalysts are actually oxides of transition elements. Manganese, chromium, copper, cobalt, iron and nickel oxides are the most active in oxidation reactions. The oxides are dispersed over a high-surface alumina or other support. One of the advantages of base-metal catalysts is preferable formation of diatomic nitrogen rather than oxides of nitrogen (NOx) during oxidation of nitrogencontaining organic compounds. The large variety of possible base-metal compositions and preparation techniques results in distinctly different catalysts, and often in misconceptions about their performance in different conditions. For example, manganese oxide catalysts can take multiple forms. Manganese dioxide (MnO₂) supported over alumina and sometimes promoted by copper oxide additives is very active, but has poor thermal resistance and quickly deactivates in the presence of small amounts of chlorine, sulfur and other poisons. However, when MnO_2 is mixed with alumina and calcined at a very high temperature, it converts to lower-valence manganese oxides and aluminates, forming a composition that shows excellent activity in VOC oxidation combined with resistance to chlorine, phosphorus and extreme heat. Commercial base-metal catalysts are typically produced as cylindrical or ring-shaped extrudates or tablets. The diameter of catalyst pellets varies widely from 2-3-mm beads or cylinders to 25-mm Raschig rings. Availability of various sizes makes it possible to customize the parameters of the catalyst bed according to the requirements of each application.

Deactivation and regeneration

A critical parameter for RTO retrofit technology is catalyst lifetime, which depends largely on poisoning and masking of active catalyst sites by volatile compounds of sulfur, halogens, silicon, phosphorus and heavy metals. Poisoning relates to selective deactivation through the reaction of active metals or metal oxides with the poison. Masking occurs when the products of poison reactions block active centers and foul catalyst pores, limiting mass transfer within the catalyst pellet. Other possible reasons for catalyst deterioration

	ТАВ	LE 1. CASE STUDY EXAN	IPLES	
Example	1	2	3	4
Industry	Composite board	Expandable polystyrene	Fiberglass insulation	Automotive part painting
Source of emission	Medium-density fiberboard dryers and press	Continuous processes and batch reactors in EPS production	Fiberglass cur- ing ovens	E-coat, powder/sealer and main- color ovens, basecoat dehydra- tion and clearcoat flash zones
VOCs	Formaldehyde, methanol, terpenes	Pentanes, styrene	Formaldehyde, methanol, phenol	Toluene, ethylbenzene, xylene, methyl ethyl ketone and other paint solvents
Gas flowrate, scfm	90,000-105,000	22,000-30,000	31,500-36,000	90,000-100,000
Adiabatic temperature rise of VOC oxidation (ΔT_{voc}), °F	~10	50-300	0.5-5	15-30
Number of canisters	5	3	3	6
Catalyst contaminants	Alkali and alka- line earth metals, sulfur dioxide	Less than 1 ppm hydro- chloric acid, small con- centrations of silicon	Sulfur dioxide, phosphorus, silicon	Inorganic compounds contained in paint pigments (tin, phospho- rus, silicon)
Inlet temperature in °F	145-155	Ambient	200-250	240-300
Maximum temperature, °F				
RTO	1,600	1,530	1,500	1,450
RCO	880	860	800	800
Outlet-inlet temperature difference, °F				
RTO	136	290	120	76
RCO	56	105	40	28
RTO thermal efficiency (η) estimated using Equation (6)	93.9%	84.2%	93.7%	95.1%
Fuel savings, million Btu/hr	9.2	5.7	3.3	5.6
VOC destruct. & removal eff.	98 %	99 %	95 %	95%
Catalyst service time, years	> 7 (actual)	10 (expected)	7 (actual)	6 (actual)

include the following:

- Fouling the pores and plugging the catalyst bed by particulate matter
- Loss of active internal surface due to sintering caused by overheating
- Mechanical destruction caused by thermal stress

• Volatilization of active component or support after the reaction with poison Rates of masking or poisoning can be vastly different depending on the particular catalyst.

Base-metal catalysts often show higher poison resistance than noble metals. For example, a study involving several catalysts exposed to 50 ppm of tetramethyl silane at 750°F showed that platinum catalysts lose their activity more quickly compared to base metals. The difference between the two base-metal catalysts, a manganese oxide and copper-chromium, correlates with the difference in the internal surface area. The copperchromium catalyst has a reaction area about six times as large, which requires more silicon for deactivation.

Platinum catalysts have rather good tolerance of sulfur dioxide. Recently developed chromium and cobalt-chromium formulations represent sulfurresistant base-metal catalysts.

One of the important qualities of commercial VOC oxidation catalysts is "regenerability." The catalyst should restore its activity after an applicable regeneration procedure, such as heating or washing. Further, it should withstand repeated cycles of regeneration without substantial deterioration in activity and mechanical strength. Regeneration through thermal treatment is easily accessible in the retrofitted RTOs.

The regeneration procedure depends on catalyst operation time. Initially, poisons or masking agents form weak bonds with the catalyst and can be removed relatively easily, for example by applying a moderate temperature increase. Upon longer operation, molecules of catalyst poisons bond more strongly with the catalyst surface; the bulk chemical or phase catalyst composition can change after the reactions with poisons, and regeneration can require higher temperatures and longer durations.

Sometimes, the operation should include gradual temperature increases in order to compensate for catalyst activity reduction. As the catalyst activity eventually approaches zero, the temperature will rise to about 1,450 °F, when VOC destruction is achieved through thermal oxidation. Fuel savings, accordingly, drops to zero compared to the original oxidizer. At some point, the plant faces a dilemma: spend an ever-increasing amount to continue operation with less and less active catalyst load, or replace the catalyst and return fuel costs to much lower levels. Figure 4 presents annualized cost return calculated at different catalyst replacement times for an RTO operating in an automobile painting operation. The optimum service time for this application was found to be between four and six years. The optimum replacement period depends on the fuel price. More expensive fuel justifies more frequent replacement.

Case examples

The following examples (Table 1) relate to RTO retrofit using ring-shaped manganese oxide catalyst (Figure 5). This catalyst is made by extrusion from a pretreated raw-material mix followed by calcination at high temperature. The size of the catalyst pellets allows a good balance between activity, pressure drop and oxidizer volume utilization for many commercial RTOs operating at linear velocities in packed beds lower than 200–250 ft/min (1–1.3 Nm/sec).

Engineered wood industry U.S. plywood, fiberboard and veneer plants commonly use large RTOs for meeting MACT (maximum achievable control technology) standards on methanol, formaldehyde and other hazardous air pollutants. In Example 1, a medium-density fiberboard mill applies a five-

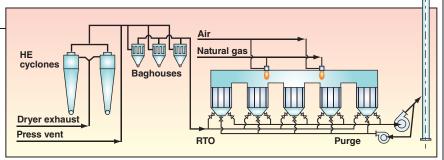


FIGURE 6. This flow diagram shows emission control equipment applied in a composite board system

canister regenerative oxidizer after a direct natural-gas-fired wood-fiber dryer and hot press. Prior to the oxidizer the particulate material emissions are removed in high-efficiency (HE) cyclones and baghouses (Figure 6). The RTO operating cycle includes canister purge by outlet gas to remove spikes of non-oxidized VOCs collected under the bed. The inlet gas flowrate to the RTO is between 90,000 and 105,000 scfm.

Before retrofit, the original RTO was operated at 1,600 °F with the difference between the outlet and inlet temperatures 136 °F. Thermal efficiency (η) estimated using Equation (6) is 93.9%. For the retrofitted unit, the temperature difference was estimated at 56°F at maximum temperature 880 °F. Accounting for a VOC oxidation temperature rise of about 10 °F, the fuel consumption would be reduced by a factor of 2.7.

The retrofitted RTO has operated for more than eight years with no change in temperature setpoint. Initial performance testing demonstrated a VOC removal efficiency of more than 97.5%, which was confirmed in further annual tests. Analyses on formaldehyde and methanol performed after 6.5 years of catalyst service have shown removal efficiencies for these components at more than 95%.

Annual catalyst tests showed that the catalyst activity decreased to some degree during the first two to three years of operation, but it remained unchanged during the following five years. The tests also showed that activity can be substantially improved after thermal regeneration. The regeneration was accomplished in the actual oxidizer after 6.5 years of operation through a gradual increase in burner firing and maintaining 1,400°F in the RTO combustion chamber over 1.5 days. Field tests confirmed the efficiency of regeneration.

The original and retrofitted systems have approximately the same electric consumption. Confirming estimates, the fuel consumption decreased by two-thirds. Natural gas savings reach about 10,000 ft³/h. Expenditures for the retrofit, including catalyst cost and installation, were justified in less than one year.

Expandable polystyrene A chemical plant produces expandable polystyrene (EPS) beads using pentanes as a blowing agent. A three-canister RTO takes about 30,000-scfm of vent air with about 400 ppm of pentanes. In addition, there is a periodic 100–500 scfm, high-VOC stream with 3–6% of pentane, directed to the RTO combustion chamber.

The original oxidizer was designed to rather small thermal efficiency $(\eta \sim 84.2\%)$. Estimates in Table 1 show possible reduction of fuel consumption in the RCO by 70–100%.

The manganese oxide catalyst (Figure 5) was selected because it could withstand very high temperatures during inflows of the high-VOC stream.

Currently, the catalyst has served in the oxidizer for more than three years. Annual testing of catalyst samples shows no decrease in catalyst activity. Expected catalyst lifetime is about ten years. Payback time for the catalyst was about five months.

Fiberglass insulation An insulation manufacturing plant uses a three-canister RTO for removing VOCs after a fiberglass curing oven. The RTO thermal efficiency determined from actual data using Equation (6) is about 93.7%. Using the catalyst saves about 67% of auxiliary fuel.

Manganese oxide catalyst was selected because of its high activity in oxidation of alcohols and aldehydes. Sulfur dioxide in the emissions gradually reduces catalyst activity through the formation of inactive manganese and aluminum sulfates. Tests showed that the sulfated catalyst can be regenerated at high temperatures. The retrofitted RTO system uses a pro-



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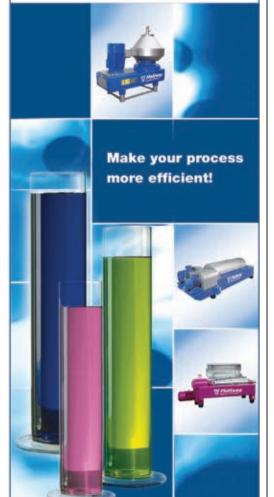


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grammed temperature increase to 1,600–1,650 °F every four months for catalyst regeneration. Currently, the unit uses a second catalyst charge with the first charge serving seven years. The payback time after catalyst installation was less than ten months.

Automotive paint booths Solvent emissions after various stages of automotive body paintings are directed to a six-canister regenerative oxidizer. The operations do not include a canister purge cycle, and, as a result, the oxidizer has relatively high thermal efficiency, $\eta = 95.1\%$. Loading the catalyst provided energy savings of about 5.6 million Btu/h. which equated to more than 60% of energy used prior to the retrofit. In this unit, the manganese oxide catalyst served more than six years.

Conclusions

Adding a catalyst over heat-exchange ceramic beds is a straightforward option for energy savings in regenerative thermal oxidizers. General estimates show that at low VOC concentrations, energy consumption can be reduced in the retrofitted RTOs by 50%-65%. In most situations, the retrofit can be accomplished easily without any changes in RTO's mechanical design and automatic control system. Additional benefits include: lower NOx and CO₂ emissions and less equipment wear-and-tear due to lower operating temperature.

Despite its simplicity, the retrofit option should be thoroughly examined for each application. The most important factors affecting the decision include: catalyst stability to the presence of trace quantities of sulfur, halogens and other contaminants: possibility for catalyst regeneration and bed burnouts without oxidizer shutdown; catalyst lifetime; bed pressure drop; and catalyst material and installation cost versus longterm operating savings.

Transition metal oxides are applied as catalysts in many retrofitted RTOs. The case studies exemplify using Mn₂O₃/Al₂O₃ catalyst, which features extreme thermal stability along with good tolerance to halogens, phosphorus and other catalyst poisons. Shaping the catalysts as large-size Raschig rings offers the opportunity for RTOs to operate at linear velocities less than 300 ft/min (1.5 Nm/sec). The catalyst lifetime can be 10 years or longer.

Catalyst performance can be monitored by testing catalyst samples taken from the operating oxidizer every one or two years. Based on the test results, the catalyst vendor or independent consultant can recommend procedures for catalyst regeneration. Gradual temperature increase may be necessary for compensating catalyst activity reduction, a technique that extends catalyst lifetime and provides additional operating cost savings.

Edited by Scott Jenkins

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Agglomeration Technology: Mechanisms

Bryan J. Ennis E&G Associates, Inc.

owder agglomeration is used in a wide variety of the chemical process industries (CPI), and a virtually endless number of process options are available. Selection requires engineers to make a substantial number of design decisions, such as the choice between wet or dry processing, the intensity of mixing and shear rates, continuous versus batch operation, cross contamination of products and ease of cleaning. These choices must be made in light of desirable agglomerate end-use properties. Key agglomeration mechanisms and their impact on agglomeration processing are reviewed in this article. The impact on process selection is also touched upon within the context of mechanisms.

Agglomeration processes

Agglomeration processes can be loosely broken down into agitation and compression methods. Agglomeration by agitation will be referred to as wet granulation. Processes include fluidbed, disc, drum, and mixer granulators, as well as many hybrid designs. Here, a particulate feed is introduced to a process vessel and is agglomerated, either batch-wise or continuously. to form a granulated product. The feed typically consists of a mixture of solid ingredients, referred to as a formulation, which includes a key active product ingredient (API), binders, diluents, flow aids, surfactants, wetting agents, lubricants, fillers and end-use aids (such as sintering aids, colors and taste modifiers). Agglomeration can be induced by a solvent or slurry atomized onto the bed of particles, or by the controlled sintering or partial melting of a binder component of the feed. Product



Granule properties (such as size, bulk density

attrition, dispersion, flowability)

(size, voidage)

f (operating variables + material variables)

+ formulation design)

(process design

Consolidation

Wetting

Breakage

FIGURE 1.

The rate mechanisms of agitation

agglomeration, or

powder wetting,

granule growth, granule consolidation and granule

attrition [1]

granulation include

forms generally range from spherical agglomerated or layered granules, to coated carrier cores.

Growth /

Q

In the second approach of agglomeration by compression, or compaction, a powder blend is fed to a compression device that promotes agglomeration due to large, applied compaction pressures. Continuous sheets of solid material are produced, as in roll pressing, or some solid form is made, such as a briquette or tablet. Continuous sheets or strands may either break down in subsequent handling to form a granulated material, or the material may be further processed through a variety of chopping, spheronizing or forced screening methods. Carrier fluids may be added or induced by melting, in which case the product is wet extruded. Compaction processes range from confined compression devices, such as tabletting, briquetting machines and ram extrusion to unconfined devices, such as roll presses and a variety of pellet mills.

In a CPI plant, an agglomeration process involves several peripheral unit operations, such as milling, blending, drying or cooling and classification, referred to generically as an agglomeration circuit. In addition, more than one agglomeration step may be present as in the case of pharmaceutical or detergent processes. In troubleshooting process upsets or product quality deviations, it is important to consider the high degree of interaction between all unit operations involved in solids processing facilities.

Agglomeration is typically used to

create free-flowing, non-segregating, uniform blends of key ingredients, with agglomerates of controlled strength that can be reproducibly metered in subsequent operations. The desired attributes of the agglomerate clearly depend on the application at hand. Still, it is important to appreciate the generic impact of agglomerate size distribution and porosity, both of which impact final product appearance. Agglomeration is used to achieve numerous benefits*. For example, a proper size distribution of granules improves solids flow, deaeration and compaction behavior, but minimizes segregation. Granule porosity controls strength, attrition resistance and dissolution rate, impacts capsule and tablet behavior, and controls surface-to-volume ratios of catalyst applications. The generic agglomeration mechanisms of granulation and compaction are addressed in the following sections.

Wet granulation

Granulation processes produce granules of low to medium, and in some cases, high density. Ranked from lowest to highest levels of shear, these processes include fluid-bed, tumbling, and mixer granulators. Four key rate mechanisms contribute to all granulation methods, as outlined by Ennis [1]. The reader is referred elsewhere for a more complete treatment [1–7]. The key rate mechanisms include wetting and nucleation, coalescence or growth,

^{*} For more information, see box titled "Objectives of size enlargement" in the online version of this article at www.che.com

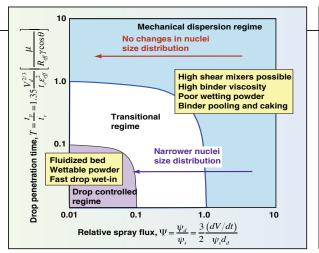


FIGURE 2. This regime map for wetting and nucleation relates spray flux, solids mixing (solids flux and circulation time) and formulation properties [3.6]

consolidation, and attrition or breakage (Figure 1). Wetting of the initial feed promotes nucleation of fine powders, or a coating for particle sizes in excess of drop size. In the coalescence or growth stage, partially wetted primary particles and previously formed nuclei coalesce to form granules composed of several particles. As granules grow, they are compacted by the forces arising from bed agitation. This consolidation stage strongly influences internal granule voidage (or porosity), and therefore end-use properties, such as granule strength, hardness and dissolution. Formed granules may be particularly susceptible to attrition if they are inherently weak or if flaws develop during drying.

Wetting. The mechanism of nucleation and wetting may be determined from a wetting regime map (Figure 2), and is controlled by two key parameters [3-6]. One is the time required for a drop to wet-in to the moving powder bed (t_p) in comparison to process circulation time (t_c) . This defines a dimensionless, drop penetration time, or $T = t_p / t_c$. The second parameter is the actual spray flux of drops (ψ_d) in comparison to solids flux moving through the spray zones (ψ_s) . This defines a dimensionless, relative spray flux of $\Psi = \psi_d / \psi_s$, which is a measure of the density of drops falling on the powder surface. If wet-in is rapid and spray fluxes are low $(T < 0.1, \Psi < 0.1)$, individual drops will form discrete nuclei in a droplet-controlled regime. At the other extreme, if drop penetration is slow and spray flux is large (T>0.5, $\Psi > 0.5$), drop overlap, coalescence and pooling of binder material will occur. Shear forces due to solids mixing then control the breakdown of wet mass clumps in a mechanical-dispersion regime, independent of drop distribution. Drop overlap and coalescence occur to a varying extent in a transitional regime, with an increasingly wider nucleation distribution being formed for increasing spray flux and decreasing wet-in time. Spray flux is strongly influenced by process and nozzle design, whereas penetration times are a strong function of the binder-powder formulation.

In Example 1 (see box, p. 36), the spray flux is close to the limit necessary to remain in a droplet-controlled regime of wetting, which forms discrete nuclei. To lower the spray flux by a factor of two, as a safety for droplet-controlled nucleation, either two nozzles spread well apart, twice the solids velocity, or half the spray rate would be needed (or, doubling the spray cycle time). Alternatively, if five times the spray rate were required, wetting would occur in the mechanical dispersion regime, diminishing the need for spray nozzles.

For a 100-fold increase in viscosity, representative of a typical binding solution and twice the drop size, the penetration time would increase to 0.4 seconds. This time is short when compared to the circulation times of high shear systems, suggesting a move toward mechanical dispersion. Drop penetration time decreases with the powder material variables of increasing pore radius, decreasing binder viscosity and increasing adhesion tension, and the operating process variables of decreasing drop size and increasing process circulation time (t_c) . Circulation time is a function of mixing and bed weight, and can change significantly with scaleup. Granule growth. There are strong interactions between the granule growth and consolidation (Figure 3). For finepowder feed, granule size often pro-

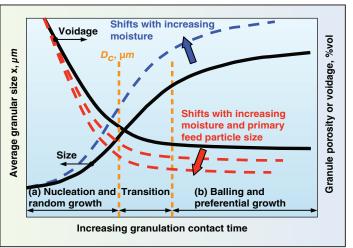


FIGURE 3. Typical regimes of granule growth and consolidation are shown [7]. Increases in growth and consolidation rate are indicated for increasing moisture and feed particle size

gresses through rapid, exponential growth in an initial nucleation stage, followed by a transition stage, finishing with very slow growth in a final balling stage [7]. In the nucleation stage, growth rate is random or independent of granule size, whereas in the balling stage, growth rate is preferentially dependent on size. While growth is occurring, granule internal porosity decreases with time as the granules are compacted. This connection between growth and densification is a dominant theme in wet granulation. Additional modes of granule size change include layering of raw material onto previously formed nuclei or granules, breakdown of wet clumps or wall buildup into stable nuclei, and rupture and attrition of wet or dry granules, respectively.

The degree of granule deformation taking place during granule collisions defines growth mechanisms (Figure 4). If little deformation takes place, the system is referred to as a lowdeformability, low-shear process. This generally includes fluid-bed, drum and disc granulators. Growth is largely controlled by the extent of any surfacefluid layer and surface deformability, which acts to dissipate collisional kinetic energy and allow permanent coalescence. Growth generally occurs at a faster time scale than overall granule deformation and consolidation. This is depicted in Figure 4, where smaller granules can still be distinguished as part of a larger granule structure. As granules are compacted, they become smoother over time due to the longer time-scale process of consolidation. This separation in time scale and interaction makes low-deformability, lowshear systems (such as fluid-beds and

EXAMPLE 1. WETTING REGIME

Feature Report

drums) easiest to scale-up and control for systems without high recycle.

For high shear rates, large granule deformation occurs during collisions, and granule growth and consolidation are intimately linked and occur on the same time scale. Such a system is referred to as a deformable-high-shear process, and includes continuous pin and plow shear mixers, as well as batch high-shear pharmaceutical mixers. In these cases, kinetic energy is dissipated through deformation of the wet mass composing the granule. Rather than the sticking-together mechanism of low deformability processes such as a fluid-bed, granules are smashed or kneaded together, and smaller granules are not distinguishable within the granule structure. High-shear, high-deformable processes generally produce denser granules than their low deformability counterpart. In addition, the combined and competing effects of granule coalescence and consolidation make high shear processes (such as mixers) difficult to scale-up with wet-mass rheology controlling granule properties, though this is still poorly understood.

Two key dimensionless groups control growth. As originally defined by Ennis [1, 8] and Tardos [9], these are the viscous and deformation Stokes numbers given respectively by:

$$St_{v} = \frac{4\rho u_{0}d}{9\mu}$$
(7)
$$St_{def} = \frac{\rho u_{o}^{2}}{\sigma_{y}} (\text{impact}) \text{ or } \frac{\rho (du_{o}/dx)^{2}d^{2}}{\sigma_{y}} (\text{shear})$$
(8)

Both numbers represent a normalized granule kinetic energy. The viscous Stokes number is the ratio of kinetic energy to viscous work due to binding fluid occurring during granule-particle collisions. Low St_v or low granule energy represents increased likelihood of granule growth, and this occurs for small granule or particle size (d), low relative collision velocity (u_{ρ}) or granule density (ρ) , and high binder phase viscosity (μ). Note d is the harmonic average of granule diameter. The deformation Stokes number (St_{def}) represents the amount of granule deformation taking place during collisions, and is a ratio of kinetic energy to wet-mass yield stress (σ_{ν}) . To illustrate wetting regime determination, consider a powder bed of width (*B*) equal to 0.10 m, moving past a flat spray with volumetric spray rate (dV/dt) equal to 100 mL/min at a solids velocity (*w*) equal to 2.5 m/s. For a given spray rate, the number of drops is determined by a drop volume or diameter (d_d) of 100 µm, which in turn defines the drop area (a_d) per unit time that will be covered by the spray, giving a spray flux (ψ_d) of:

$$\psi_{d} = \frac{da_{d}}{dt} = \frac{dV/dt}{V_{d}} \frac{\pi d_{d}^{2}}{4} = \frac{3}{2} \frac{(dV/dt)}{d_{d}} = \frac{3}{2} \frac{(100 \times 10^{-6}/60 \ m^{3/s})}{(100 \times 10^{-6} \ m)} = 0.025 \ m^{2}/s$$
(1)

Where V_d is the drop volume.

As droplets contact the powder bed at a certain rate, the powder moves through the spray zone at its own velocity, or at the solids flux (ψ_s) . The solids flux and the dimensionless, relative spray flux (Ψ) are then given for this simple example by:

$$\psi_s = \frac{dA}{dt} = Bw = 0.10 \ m \times 2.5 \ m/s = 0.25 \ m^2/s$$

$$\Psi = \frac{\psi_d}{\psi_s} = \frac{0.025 \ m^2/s}{0.25 \ m^2/s} = 0.10$$
(2)
(3)

Where A is the spray area, B is the spray width, w is the solids surface velocity, and ψ_d and ψ_s are the drop and solids fluxes respectively.

For a lactose powder of surface-to-volume average diameter of $d_{32} = 20 \ \mu m$, and loose packing and tapped packing voidage of $\varepsilon = 0.60$ and $\varepsilon_{tap} = 0.40$, the effective voidage and pore radius are given by:

$$\varepsilon_{eff} = \varepsilon_{tap} \left(1 - \varepsilon + \varepsilon_{tap} \right) = 0.4 \left(1 - .6 + .4 \right) = 0.32 \tag{4}$$

$$R_{eff} = \frac{\Phi \, d_{32}}{3} \left(\frac{\varepsilon_{eff}}{1 - \varepsilon_{eff}} \right) = \frac{0.9 \times 20}{3} \left(\frac{0.32}{1 - 0.32} \right) = 2.8 \,\mu m \tag{5}$$

Where ε is the loose effective packing voidage; ε_{tap} is the tapped packing voidage; ε_{eff} is the effective voidage; d_{32} is the average diameter; φ is the particle sphericity; and R_{eff} is the effective pore radius.

For droplet-controlled growth, a short drop wet-in or penetration time is required, and should be no more than 10% of the circulation time (t_c). For water with a viscosity of 1 cP (0.001 Pa-s), and an adhesion tension of .033 N/m, we obtain a penetration time of:

$$t_{p} = 1.35 \frac{V_{d}^{2/3}}{\varepsilon_{eff}^{2}} \left[\frac{\mu}{R_{eff} \gamma \cos \theta} \right] = 1.35 \frac{\left(\pi \left(100 \times 10^{-6} \right)^{3} / 6 \right)^{1/5}}{0.32^{2}} \left[\frac{0.001}{2.8 \times 10^{-6} \times .033} \right] = .0009 \ s$$
(6)

Where μ is the binder viscosity; γ is the binder surface tension; θ is the contact angle; $\gamma \cos \theta$ is the adhesion tension; and t_p is the drop wet-in penetration time.

Note that granule collisional velocities require judicious estimation.

Examples of growth behavior are illustrated with the help of Figure 5. In the limit of low deformability $(St_{def} = 0)$, growth is controlled by St_v and bed moisture. This includes fluidized beds, discs and drums. For low St_{i} less than some critical value, initial growth rate becomes a function of moisture and mixing only as illustrated for the (a) nucleation-random growth stage (Points 1 to 3, black curve). To a first approximation, the initial growth behavior is actually independent of St_{v} , granule inertia and binder viscosity. This growth curve may vary from linear at small spray rates, as in fluid beds, to exponential growth in drums or at high moisture levels. Increases in spray rate (or more generally spray flux), bed moisture, and mixing (such as drum rotation rate, or fluid-bed excessgas velocity when ignoring attrition) increase initial growth rate. The width of the granule size distribution typically increases in proportion to average size (Figure 5, inset a). An exception would be disc granulation, which possesses self-granule size classification.

Later in the (b) balling/preferential growth stage (Figure 5, Points 4 and 5, black curve), granules will reach some limiting size. Here, the granule size distribution will narrow and continue to grow only by layering of powder and smaller granules onto granules in excess of the limiting size (Figure 5, inset b). This final limit increases with decreasing St_v (shown in dashed blue), or increases with increasing binder viscosity, and decreasing granule inertia (see Example 2 on p. 38). Granule attrition will also contribute to the final growth curve.

For deformable, high shear processes, namely high shear mixers, initial growth rate increases with

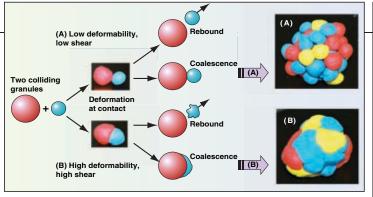


FIGURE 4. Granule structures resulting from (A) low and (B) high deformability systems are typical for fluid-bed and high-shear mixer granulators, respectively [4]

increasing deformation Stokes number (St_{def}) , as illustrated in Figure 5 (shown in red), representing an increased kneading together of granules in the process. This occurs for increasing impeller speed, granule density, and decreasing formulation yield stress (σ_{ν}). Yield stress generally decreases with increasing bed moisture (or saturation), increasing particle size or pore radius, decreasing binder viscosity, and decreasing surface tension. As granules densify in a high shear process, their yield stress rises and they become less deformable which works to lower coalescence in the later stages of growth — and a limit of growth will again be achieved as with St_v . Often this limiting size will vary inversely with the initially observed growth rate.

Lastly, it should be noted that the process or formulation itself cannot uniquely define whether it falls into a low or high deformability category. A very stiff formulation with low deformability may behave as a high deformability system in a high shear mixer, or a very pliable formulation may act as a low deformability system in a fluid-bed granulator.

Consolidation. Granule consolidation or densification is also controlled by Stokes numbers and peak bed moisture. Consolidation typically increases for all processes with increasing residence time, shear levels, bed height, bed moisture or granule saturation, particle feed size or pore radius, surface tension, and decreasing bindingfluid viscosity. The roles of moisture, feed particle size and processing time are illustrated in Figure 3. Simultaneous drying or reaction usually acts to arrest granule densification.

Attrition. Breakage and attrition play critical roles in defining a final agglomerated product quality as well as final strength attributes for subsequent processing and handling [1-4, 10]. Attrition is controlled by a combination of granule voidage and inherent bond strength, which may be assessed by mea-

surements of fracture toughness and hardness on prepared bar composites of the formulation. In addition, direct indentation measurements of hardness of granules and particles are possible in some cases. Granule or agglomerate voidage is controlled by the mechanism of consolidation, with denser granules giving less attrition and requiring greater work for re-dispersion in solution.

Granulation processes

The granulation mechanisms described above can occur simultaneously in all wet granulation processes, and they determine the final granule size distribution, voidage and the final product quality. However, certain mechanisms may dominate in a particular process. It is vital to keep in mind the high degree of interaction between formulation properties and process equipment in making a selection of process equipment.

With small shear rates and simultaneous drying, batch fluid-bed granulators can produce some of the lowest density granules, and are an example of low deformable growth. Growth rate is controlled primarily by the wetting process, spray rate and current bed moisture. Low spray fluxes and fast drop penetration are required to prevent binder pooling and defluidization. Poorly wetting powders or binders of initially high viscosity are precluded. Consolidation of granules can be increased independent of growth through increasing bed height, bed moisture or process residence time. The inherent stability of

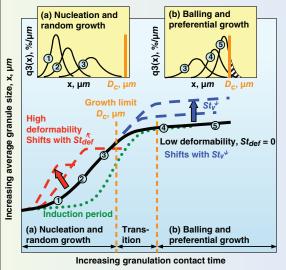


FIGURE 5. Typical granule-growth profiles for low and high deformability mechanisms are shown. The insets illustrate the corresponding evolution of size distribution, above and below the growth limit D_c [4]

low deformability processes allows a wide manipulation in granule properties, as well as ease of scaleup.

At the other extreme are high shear mixer granulators, where mechanical blades and choppers induce binder distribution and growth, producing medium to dense, sometimes irregular granules. Mixers generally operate as a deformable growth process, where in most cases it is difficult to control granule density independent of size. Mixers have an advantage in that they can process plastic, sticky or poorly wetting materials, and can spread viscous binders while operating in a mechanical dispersion regime of nucleation. However, associated with this flexibility in processing a wide variety of materials, high shear mixers can be very difficult to scale-up due to large shifts in the competition between growth and densification, wetting regimes, and powder mixing with vessel scale.

Tumbling granulators produce spherical granules of low to medium density, and lie between fluid-bed and mixer granulators in terms of shear rate and granule density. They have the highest throughput of all granulation processes. In the case of drums, processes often operate with high recycle ratios, whereas preferential segregation in disc granulators can produce very tight size distributions of uniform spherical granules.

Compaction and extrusion

Compressive techniques of agglomeration range from completely confined compaction processes, as in case of tabletting, to unconfined as in the case

EXAMPLE 2. DRUM GRANULATION

For the drum granulation of ground iron ore feed ($d = 5 \mu m$, $\rho = 3 \text{ g/cm}^3$) and water ($\mu = 0.01 \text{ P}$) at a rotation rate (N) of 10 rpm, the initial starting collisional velocity (u_o) by relative shear and the viscous Stokes number are given by:

$$u_{o} = \omega d = \frac{2\pi N}{60} d$$

$$= \frac{2\pi 10}{60} (5 \times 10^{-4}) = 5.2 \times 10^{-4} \ cm/s$$
(9)

$$St_{\nu} = \frac{9\,\mu}{9\,\mu}$$

$$= \frac{4 \times 3 \times 5.2 \bullet 10^{-4} \times 5 \bullet 10^{-4}}{9(.01)} \approx 3.5 \times 10^{-5}$$
(10)

Where ω or N is the rotation rate; d is the average granule or particle diameter; ρ is the granule or particle density; and μ is the binder viscosity.

For such a low value of $St_v << 1$, all the iron ore particles will adhere to one another provided local binding water is present. As granulation continues, diameter will increase along the growth curve given in Figure 7, with the rate controlled by moisture level and rotation rate (or solids residence time). This occurs until a transition limit of size is reached where St_v approaches one. In this example, the limit is about 1 mm, after which growth of these granules continues by balling. In the presence of binder such as bentonite clay, larger diameters are possible, as this will raise the effective viscosity of the binding solution. Alternately, lowering drum speed will give a larger limit, but will decrease the initial rate of growth.

of roll pressing [2-4]. Due to the importance of powder friction and compression, we also include here wet extrusion techniques, such as screw extrusion and pellet-type mills. The success of these unit operations is determined by the ability of powders to freely flow, uniformly transmit stress, readily deaerate, easily compact forming permanent interparticle bonding, and maintain bonding and strength during stress unloading (Figure 6). These mechanisms are controlled in turn by the geometry of the confined space, the nature of the applied loads and the physical properties of the particulate material and of the confining walls.

Powder filling and compact weight variability are strongly impacted by bulk density control and powder flowability, as well as any segregation tendencies of the feed [11-12]. In the case of unassisted flow of free-flowing coarse material through an orifice of diameter *B*, the mass discharge rate is given by the Beverloo relation:

$$W_{o} = 0.58 \rho_{b} \sqrt{g} \left(B - kd_{p} \right)^{2.5}$$
 (11)

Here, ρ_b is bulk density, k equals 1.5 for spherical particles, and d_p is particle size. This is a maximum achievable filling rate, and it is a strong function of opening size, B. Carefully controlling gap distances for free flowing materials is critical. In practice,

mass feedrate can decrease substantially with increasing powder and wall friction, increasing powder cohesion, decreasing bulk-powder permeability, and decreasing opening size (small compacts). Reproducible powder feeding is crucial to the smooth operation of compaction techniques. High powder cohesion and low permeability can lead to wide feed fluctuations and in the worst case, can entirely arrest the flow. In fact, permeability plays a large role in determining maximum production rate in compaction processes.

Following the filling of a compression zone, stresses are applied to the powder with the aim of forming interparticle bonds. However, the frictional properties of powders prevent a uniform stress transmission. For a given applied load, wide distributions in local pressure and the resulting density can exist throughout the compact [13] as illustrated in Figure 7. The axial (σ_z) and radial (σ_r) stresses decrease exponentially with axial distance, z, from the applied load, σ_o .

The ratio σ_z/σ_0 may be taken as a measure of stress uniformity, which in practice increases toward unity for decreasing aspect ratio of the compact, decreasing diameter, increasing powder friction, and most important, decreasing wall friction, as controlled by the addition of lubricants (Figure 7 and Example 3). Low stress trans-

EXAMPLE 3. STRESS TRANSMISSION

By way of example for an aspect ratio z/D equal to one for a cylindrical compact, with an effective powder friction δ equal to 40 deg and wall friction φ' at 15 deg, let us determine the percentage of stress transmitted from an applied load using Janssen's relation:

$$K = (1 - \sin \delta) / (1 + \sin \delta)$$

= $(1 - \sin 40^\circ) / (1 + \sin 40^\circ) = 0.643$ (12)

$$\mu_w = \tan \phi' = \tan \left(15^\circ \right) = 0.268 \tag{13}$$

$$\frac{\sigma_z}{\sigma_o} = e^{-4\mu_w K(z/D)}$$

$$= \exp(-4 \times 0.268 \times 0.643 \times 1) = 0.79$$
(14)

Where K is the lateral Janssen constant; μ_w is the wall friction coefficient; D is the die diameter; z is the axial distance from the applied load; and σ_z/σ_o is the ratio of axial to applied stress.

We obtain a ratio of top-applied punch stress to bottom punch stress of 79% when pressing from one side. Twenty percent of this stress is lost to die wall friction, which could result is large density variations (Figure 7) and delamination during unloading. In this case, a decrease in wall friction (φ') to 3 deg due to lubricants gives an approved stress ratio of 96%.

> mission results in poor compact uniformity, unnecessarily large compression loads to compact weak zones, and large residual radial stresses after stress unloading, giving rise to flaws and delamination as well as large die ejection forces.

> For a local zone of applied stress, particles deform at their point contacts, including plastic deformation for forces in excess of the particle surface hardness. This allows intimate contact at surface point contacts, allowing cohesion and adhesion to develop between particles, and therefore interfacial bonding, which is a function of their interfacial surface energy. Both particle size and bond strength control final compact strength for a given compact density or voidage. While brittle fragmentation may also help increase compact density and points of interparticle bonding as well, in the end some degree of plastic deformation and interlocking is required to achieve some minimum compact strength. Successful compaction requires that a minimum critical yield pressure be exceeded to obtain significant strength. This yield pressure increases linearly with particle hardness. Strength also increases linearly with compaction pressure, with a slope inversely related to particle size.

> During the short time scale of the applied load, any entrapped air must es-

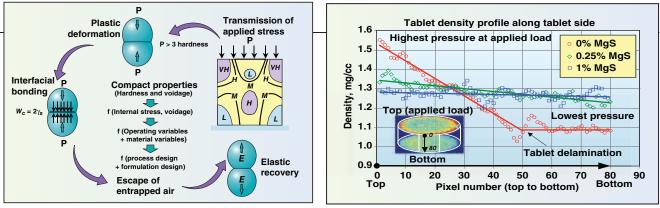


FIGURE 6. The micro-level mechanisms of compaction are shown here [4] (letters in inset refer to levels of pressure; low, high, medium and very high)

FIGURE 7. The density along a lactose tablet edge (pixel number) is shown as a function of level of magnesium stearate lubricant. The inset photo is a near infrared (NIR) chemical image of a lactose tablet density [13]

cape and a portion of the elastic strain energy must be converted into permanent plastic deformation. The developed air pressure will vary inversely with permeability, and increase with compact size and production rate. Low powder permeability and entrapped gas may act to later destroy permanent bonding, and generally lower allowable production rates.

Upon stress removal, the compact expands due to elastic recovery of the matrix, which is a function of elastic modulus and expansion of any remaining entrapped air. This can result in loss of particle bonding and flaw development, which is exacerbated for variation in compaction stress due to poor stress transmission. The final step of stress removal involves compact ejection, where any remaining radial elastic stresses are removed. If recovery is substantial, it can lead to capping or delamination of the compact. Therefore, most materials have an allowable compaction pressure range, with a minimum pressure set by hardness, and a maximum by elastic and permeability effects.

In the case of extrusion, both wet and dry techniques are strongly influenced by the frictional properties of the particulate phase and wall. In wet extrusion, wet mass rheology and friction control the pressure needed to in-

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duce die extrusion, with this pressure increasing with desired throughput. On the other hand, the actual pressure that can be developed by the sliding action of the barrel from the reference frame of the screw flight decreases with increasing throughput and screw friction, and increases with decreasing barrel friction. Lastly, the rheological properties of the liquid phase are equally important. Poor rheology can lead to separation of the fluid and solid phases, large rises in pressure, and undesirable sharkskin-like surface appearance on the granulate, which is prone to high attrition [3, 4].

These mechanisms of compaction control the final flaw and density distribution throughout the compact, whether it is a roll-pressed, extruded or tabletted product; and as such, control compact strength, hardness, strength characteristics and dissolution behavior. Process performance and developed compaction pressures in extrusion and dry compaction equipment are very sensitive to powder flow and mechanical properties of the feed. These processes generally produce much denser compacts or agglomerates than wet granulation.

Process equipment selection

The choice of agglomeration equipment is subject to a variety of constraints.

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Ideally, the choice of equipment should be made on the basis of the desired final product attributes. Agglomerate porosity is a very important consideration in that it impacts strength and attrition resistance, hardness, internal surface area, reactivity and dissolution rate. The desired agglomerate appearance and size distribution, as well as the ability to utilize moisture or solvents are additional considerations. Wet granulation produces low- to medium-density granules of varying sphericity. Binders are typically utilized, and drying of solvents is required, with the associated energy and dust-air handling costs. If denser agglomerates are required, dry compaction or wet extrusion should be considered, although it is worth noting that reasonably dense granules are possible with two-stage mixer processing. Dry compaction is suitable for moisture sensitive materials. Appearance considerations might suggest tabletting, or wet granulation or extrusion combined with spheronizing for free-flowing, nearly spherical granules.

Edited by Dorothy Lozowski

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Engineering Practice

Purifying Coke-Cooling Wastewater

A new method for treating coke-cooling wastewater in a delayed coking unit

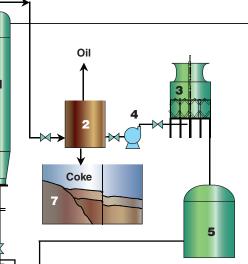


FIGURE 1. Typical coke-cooling wastewater treatment system. 1–coke drum; 2–gravity settling pit; 3–cooling tower; 4–hot coke-cooling water pump; 5–water storage tank; 6–high-pressure pump; 7–coke settling pit

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he delayed coking process is a fundamental part of the petroleum refining and petrochemical industries, and is among the main technical means for transforming inexpensive heavy oil into more valuable, light-oil products. Delayed coking units are common in petroleum refineries, and the process has been developed significantly as a heavy oil processing method in many countries, including China and the U.S.

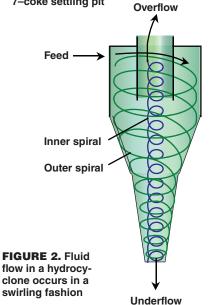
The importance of delayed coking and its advanced development has not, however, eliminated certain disadvantages. The process uses highsulfur heavy oil as its raw material, and requires a large amount of cooling water. This wastewater is referred to as "coke-cooling wastewater." The large volume of wastewater produced by a delayed coking process at a typical petroleum refinery not only contains solid coke breeze (residue from screening heat-treated coke) and liquid heavy oil, but also contains organic and inorganic sulfides - all potential sources of environmental pollution.

The problem of cleaning coke-cooling wastewater has vexed engineers for decades, but effective solutions have remained elusive. Without a lowcost technique to treat coke-cooling wastewater, environmentally inocuous production of petroleum is impossible. The process discussed here represents an improved method for purifying coke-cooling wastewater and offers a path toward cleaner petrochemical production.

Delayed coking

Essentially, delayed coking is a hightemperature process involving extensive use of direct heat to generate higher-value products from crude oil. The coking process combines severe thermal cracking and condensation reactions, and requires a large amount of high-grade energy. The process employs a heater designed to raise temperatures of the residual feedstock above the coking point without significant coke formation. The term "delayed coking" is derived from the fact that an insulated coke drum is provided for the heater effluent, so that sufficient time is allowed for coking to occur before subsequent processing.

In delayed coking, heater effluent flows into the coke drum in service. When the drum is filled to within a safe margin of capacity, the heater effluent feed is switched to an empty coke drum. The full drum is then isolated, steamed to remove hydrocarbon vapors, and filled with cooling water. Next, the drum is opened, drained and emptied, yielding a petroleum coke product. All the coke-cooling wastewater produced by the delayed coking process normally drains from the coke solids and is collected and recycled for drilling and drum cooling. A water-flow sche-



matic diagram is shown in Figure 1.

A de-oiling and clarifier system may be used before pumping water back to its storage tank. A cooling tower is used to lower the temperature of the coke-cooling wastewater. The oil and coke removal has traditionally been achieved by a gravity settling pit, but the gravity-separating efficiency is low at temperatures of around 85–125°C.

Water containing fine coke and oil can cause costly operational and maintenance problems. Solids can settle in the water storage tank, requiring routine flushing or cleaning. The concentration of fine coke then becomes abrasive and can damage the high-pressure pump, coke drilling tools, as well as the control and water-isolation valves. Damage to the pump and valves can cause a unit outage or shutdown,

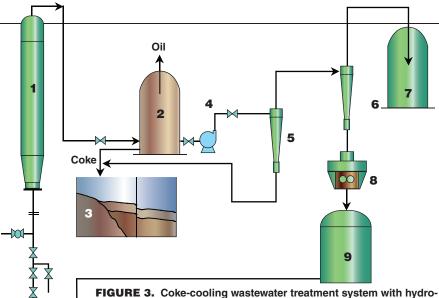


FIGURE 3. Coke-cooling wastewater treatment system with hydrocyclone. 1–coke drum; 2–hot coke-cooling wastewater tank; 3–coke settling pit; 4–hot coke-cooling water pump; 5–solid-liquid separating hydrocyclone; 6–deoiling hydrocyclone; 7–oil tank; 8–air cooler; 9–water storage tank; 10–high-pressure pump



FIGURE 4. A de-oiling hydrocyclone is shown with an air cooler in a plant facility

which raises costs significantly. Since the system is open to the atmosphere, the oil and sulfides in the cooling tower wastewater become environmental pollutants via evaporation.

Separating phases

To move past the difficulties associated with purifying wastewater from delayed coking processes, we have designed a closed process that offers a reliable and cost-effective method to treat wastewater from industrial cool-coking. The method comprises the following steps (Figure 2): cooling the wastewater mixture produced from delayed coking to between 5 and 50°C; subjecting the cooled wastewater to a solid-liquid separation step to obtain a coke breeze phase and a liquid phase; further separating the resulting liquid phase into oil and water phases; and discharging water from the oil phase.

The solid-liquid hydrocyclone separator (Figures 3-5) is deployed between a coke-cooling hot wastewater tank and an oil-water separator. The coke-cooling wastewater is pumped into the hydrocyclone, wherein a majority of solid fine coke is separated from the coke-cooling wastewater at separation efficiencies of up to 70-80%. Under stable operating conditions, the separated fine coke is recycled back to the gravity settling pit for recovery. After the separated water phase is cooled by the air cooler to below 55°C, according to engineering requirements, or preferably lower than 50°C,



FIGURE 5. A solid-liquid separating hydrocyclone helps prevent sulfur pollutants

then is pumped into the coke drum.

After the separated oil phase (heavy oil) enters into an oil tank for further purification, it either returns to the coke tower for reprocessing, or it is stored in the oil tank to be pumped into the coke tower for eventual oil refining when needed.

Hydrocyclone treatment

A key component of the process is a single-stage, high-efficiency de-oiling hydrocyclone, a centrifugal separation device that, unlike other centrifugal machines, has no moving parts. The driving force for the separation comes from transforming the static energy of the fluid (fluid pressure) into dynamic energy (fluid velocity). Because of considerable research and development effort in this area, hydrocyclones are now widely used in various industries to separate two components of different densities. The devices were originally applied to particle-liquid separations, and have been used more recently for liquid-liquid and air-liquid separation as an alternative to gravity-based conventional separators. Hydrocyclones have several advantages that have led to wide industry acceptance. These include the equipment's ease of operation, capability of generating high throughput, and requirements for less maintenance and floor space.

The hydrocyclone consists of cylindrical and conical components. The liquid with suspended particles is injected tangentially through an inlet opening in the upper part of the cylindrical section. As a result of the tangential entry, a strong swirling motion develops within the hydrocyclone device.

cooled by the air cooler to below 55°C, according to engineering requirements, or preferably lower than 50°C, it enters the water storage tank, and store the hydrocyclone, centrifugal forces

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FIGURE 6. A de-oiling hydrocyclone helps improve coke-cooling efficiency

accelerate particles toward the walls. The fluid's spiral movement forces large or dense particles against the wall of the cyclone and they migrate downward toward the underflow. Fine, or low-density, particles are swept into a second inner spiral, which moves upward to the overflow. Figures 4-6 show the installation of the hydrocyclone and air cooler in the treatment process for coke-cooling wastewater.

Advantages

The method and equipment of the new system have a host of advantages over previous treatment systems for coke-

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mechanical reliability and development of novel heat transfer equipment



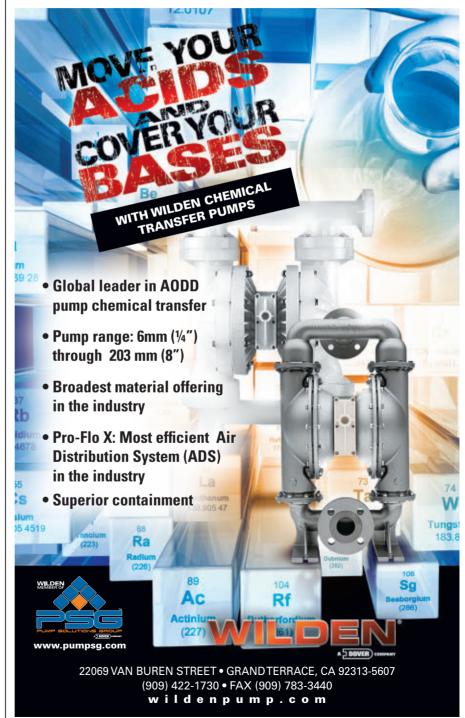
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cooling wastewater. Among them is the prevention of sulfur pollution and foul odor while purifying wastewater. The new system also saves energy and cost resources by improving the efficiency of cool coking. The system also has the potential to reduce unit outage and shutdown time.

Utilizing hydrocyclones in coke-

cooling wastewater operations helps to solve several long-standing problems associated with purifying the coke-cooling hot wastewater produced in cool coking processes. The method enables cleaner production of petrochemicals through a closed system with pollution-free discharge.

Edited by Scott Jenkins



Engineering Practice

Solubility of Water in Benzene Derivatives

Solubility and Henry's Law constants for water in benzene and its derivatives

Carl L. Yaws and Manish Rahate, Lamar University

he solubility of water in the hydrocarbons that comprise crude oil is an increasingly important issue in view of processing, safety and environmental considerations surrounding product quality and equipment sustainability. Water in the reacting hydrocarbon species may result in the formation of undesirable byproducts. The presence of water in the product may be detrimental to quality. Water content at high enough levels in the products of a given hydrocarbon process may render the products unuseable by the customer. When chemical processes involve lowering temperatures to values near the freezing point of water, solids (ice or hydrates) can form. Such solid formation will affect both the fluid flow in piping as well as the operational characteristics of process equipment. For catalytic reactions, water in the hydrocarbon may poison the catalyst that promotes the desired reaction.

To illustrate the importance of water solubility in hydrocarbons, we offer the results of solubility studies involving water in benzene derivatives. The results for both solubility and Henry's Law constant for water are provided in an easy-to-use tabular format for a wide variety of benzene derivatives contained in crude oil.

A new correlation for solubility of water is also presented. It provides reliable solubility values down to low concentrations (parts per million range). The correlation is based on the boiling point temperature of the hydrocarbon. Correlation values and experimental data agree favorably.

Correlation for water solubility In earlier work by Yaws and coworkers [22, 25], the solubility of hydrocarbons and other chemicals in water was correlated as a function of the boiling point of the compound. In this present work, it was determined that the boiling point method was also applicable for correlation of solubility of water in benzene derivatives:

$$\log_{10}(S) = A + B \times T_B \tag{1}$$

where:

- S = solubility of water in compound at 25°C, ppm by weight
- T_B = boiling point temperature of compound (K)

A = 3.780

 $B = -2.720 \times 10^{-3}$

The correlation applies to boiling point temperatures in a range from about 280K to 590K.

The coefficients (A and B) for the correlation were determined from regression of the available data. In preparing the correlation, a literature search was conducted to identify data source publications [1-25]. Excellent compilations by Englin and others [4], Glasoe and Schultz [5], Jones and Monk [6], Polak and Lu [9], IUPAC [10-14] and Sorensen and Artl [16]were consulted for solubility of water. The compilation by Yaws [23] was used for boiling point temperature. The publications were screened and copies of appropriate data were made. These data were then keyed into the computer to provide a database for which experimental data are available. The database also served as a basis to check the accuracy of the correlation.

The solubility of water versus boiling point temperature of compounds is

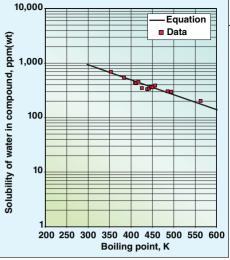


FIGURE 1. Solubility correlation with boiling points for benzene derivatives.

shown in Figure 1 for benzene derivatives. The data of Englin [4] and Polak and Lu [9], which are applicable at ambient temperature, were selected for the graph. The graph reveals favorable agreement between correlation values and experimental data.

Henry's Law constant

The results for solubility of water and Henry's Law constant are given in Table 1. In the tabulation, the results for Henry's Law constant are based on water solubility and vapor pressure at ambient conditions using the appropriate thermodynamic relationships [22]. A compilation by Yaws [24] was used for vapor pressure. The presented values are applicable for water in a wide variety of benzene derivatives (normal and branching).

The tabulated values are based on both experimental data and estimates. In the absence of data, the estimates for isomers and large-sized compounds (compounds larger than C_{10}) should be considered rough values, useful for initial analysis. If initial analysis is favorable, follow-up experimental determination is recommended.

The results are presented in a tabular format, which is especially applicable for rapid engineering usage with the personal computer or hand calculator. The tabulation is arranged by carbon number (C_6 , C_7 , C_8 ...). This provides ease-of-use in quickly locating data using the chemical formula.

Applying the data

The results for solubility and Henry's Law constant are useful in engineering applications involving water in benzene derivatives. Examples are shown below.

			TABLE 1. SOLUBILITY	OF WATE	R IN BEI	NZENE D	ERIVATI	/ES			
						ty of Wate er million			Henry's La	w Constant (H	H)
No.	ID	FORMULA	NAME	CAS No.	Т _в (К)	1	S @25°C, ppm (mol)	code	H @ 25°C, atm/mol frac	H @ 25°C, atm/mol/m ³	code
1 2	7339 11216	C ₆ H ₆	benzene	71-43-2 108-88-3	353.24 383.78	691.88 541.78	3000.00 2771.00	1	10.45 11.31	1.8804 × 10 ⁻⁴ 2.0358 × 10 ⁻⁴	.,_
2	14332	C ₇ H ₈ C ₈ H ₁₀	toluene ethylbenzene	100-00-3	409.35	441.01	2599.00	1	12.06	2.0358 × 10 ⁻⁴	
4	14333	C ₈ H ₁₀	<i>o</i> -xylene	95-47-6	417.58	454.93	2681.00	1		2.1042 × 10 ⁻⁴	
5	14334	C ₈ H ₁₀	<i>m</i> -xylene	108-38-3	412.27	431.17	2541.00	1	12.33	2.2201 × 10 ⁻⁴	- / -
6	14335	C ₈ H ₁₀	<i>p</i> -xylene	106-42-3	411.51	438.97	2587.00	1		2.1806 × 10 ⁻⁴	
7 8	17591 17592	C ₉ H ₁₂ C ₉ H ₁₂	cumene <i>m</i> -ethyltoluene	98-82-8 620-14-4	425.56 434.48	347.73 396.47	2320.00 2645.23	1	13.51 11.85	2.4316 × 10 ⁻⁴ 2.1326 × 10 ⁻⁴	
9	17593	C ₉ H ₁₂	<i>o</i> -ethyltoluene	611-14-3	438.33	387.03	2582.21	2	I	2.1847 × 10 ⁻⁴	-
10	17594	C ₉ H ₁₂	<i>p</i> -ethyltoluene	622-96-8	435.16	394.79	2633.99	2	11.90	2.1417 × 10 ⁻⁴	
11	17595	C ₉ H ₁₂	1,2,3-trimethylbenzene	526-73-8	449.27	361.40	2411.21	2	13.00	2.3396 × 10 ⁻⁴	
12 13	17596 17597	C ₉ H ₁₂ C ₉ H ₁₂	1,2,4-trimethylbenzene	95-63-6 25551-13-7	442.53	376.98 371.19	2515.17 2476.56	2		2.2429 × 10 ⁻⁴ 2.2779 × 10 ⁻⁴	
14	17598	C ₉ H ₁₂	trimethylbenzene mesitylene	108-67-8	437.89	332.74	2220.00	1		2.5411 × 10 ⁻⁴	-
15	17599	C ₉ H ₁₂	propylbenzene	103-65-1	432.39	401.70	2680.09	2	11.69	2.1049 × 10 ⁻⁴	
16	20487	C ₁₀ H ₁₄	butylbenzene	104-51-8	456.46	387.89	2890.00	1		1.9520 × 10 ⁻⁴	- / -
17	20488	C ₁₀ H ₁₄	isobutylbenzene	538-93-2	445.94	369.01	2749.35	2		2.0519 × 10 ⁻⁴	
18 19	20489 20490	C ₁₀ H ₁₄	sec-butylbenzene	135-98-8 98-06-6	446.48	370.58 339.71	2761.00	1	11.35 12.38	2.0432 × 10 ⁻⁴ 2.2289 × 10 ⁻⁴	
20	20490	C ₁₀ H ₁₄ C ₁₀ H ₁₄	cymene	25155-15-1	442.30	360.88	2688.73	2		2.2289 × 10 ⁻⁴	
21	20492	C ₁₀ H ₁₄	<i>m</i> -cymene	535-77-3	448.23	363.76	2710.20	2	11.56	2.0815 × 10 ⁻⁴	
22	20493	C ₁₀ H ₁₄	<i>o</i> -cymene	527-84-4	451.33	356.77	2658.09	2	11.79	2.1223 × 10 ⁻⁴	
23	20494	C ₁₀ H ₁₄	<i>p</i> -cymene	99-87-6	450.28	359.17	2676.00	1		2.1081 × 10 ⁻⁴	-
24 25	20495 20496	C ₁₀ H ₁₄	1-methyl-2-propylbenzene	1074-17-5 1074-43-7	457.95 454.95	342.28 348.77	2550.13 2598.50	2		2.2122 × 10 ⁻⁴ 2.1710 × 10 ⁻⁴	
26	20490	C ₁₀ H ₁₄ C ₁₀ H ₁₄	1-methyl-3-propylbenzene 1-methyl-4-propylbenzene	1074-45-7	456.45	345.51	2574.20	2		2.1915 × 10 ⁻⁴	
27	20498	C ₁₀ H ₁₄	(\$)-(1-methylpropyl)benzene	5787-28-0	457.10	344.11	2563.76	2	12.22	2.2004 × 10 ⁻⁴	1,2
28	20499	C ₁₀ H ₁₄	diethylbenzene	25340-17-4	455.05	348.55	2596.87	2		2.1723 × 10 ⁻⁴	
29 30	20500 20501	C ₁₀ H ₁₄	o-diethylbenzene	135-01-3	456.61	345.16 350.21	2571.62	2	12.19 12.01	2.1937 × 10 ⁻⁴	
30	20501	C ₁₀ H ₁₄ C ₁₀ H ₁₄	<i>m</i> -diethylbenzene <i>p</i> -diethylbenzene	141-93-5 105-05-5	454.29 456.94	350.21	2609.26 2566.31	2	-	2.1620 × 10 ⁻⁴ 2.1982 × 10 ⁻⁴	
32	20503	C ₁₀ H ₁₄	3-ethyl- <i>o</i> -xylene	933-98-2	467.11	323.19	2407.95	2	13.02	2.3428 × 10 ⁻⁴	
33	20504	C ₁₀ H ₁₄	4-ethyl-o-xylene	934-80-5	462.93	331.76	2471.82	2	12.68	2.2822×10^{-4}	1 -
34	20505	C ₁₀ H ₁₄	2-ethyl-m-xylene	2870-04-4	463.19	331.23	2467.80	2		2.2860 × 10 ⁻⁴	
35 36	20506 20507	C ₁₀ H ₁₄ C ₁₀ H ₁₄	4-ethyl- <i>m</i> -xylene 5-ethyl- <i>m</i> -xylene	874-41-9 934-74-7	461.59 456.93	334.56 344.47	2492.65 2566.48	2	12.57 12.21	2.2632 × 10 ⁻⁴ 2.1981 × 10 ⁻⁴	<u> </u>
37	20508	C ₁₀ H ₁₄	2-ethyl- <i>p</i> -xylene	1758-88-9	459.98	337.95	2517.92	2	12.45	2.2405 × 10 ⁻⁴	-
38	20509	C ₁₀ H ₁₄	1,2,3,4-tetramethylbenzene	488-23-3	478.19	301.52	2246.52	2	13.95	2.5111 × 10 ⁻⁴	
	20510		1,2,3,5-tetramethylbenzene	527-53-7		315.12	2347.79	2	1 1	2.4028 × 10 ⁻⁴	
40 41	20511 22958	C ₁₀ H ₁₄ C ₁₁ H ₁₆	1,2,4,5-tetramethylbenzene pentylbenzene	95-93-2 538-68-1	469.99 478.61	317.41 300.73	2364.91 2474.77	2	13.25 12.66		
42	22959	C ₁₁ H ₁₆	2-phenylpentane	2719-52-0	463.15	331.31	2726.38	2	11.50	2.0692 × 10 ⁻⁴	1
43	22960	C ₁₁ H ₁₆	3-phenylpentane	1196-58-3	464.15	329.24	2709.36	2	11.57	2.0822×10^{-4}	
44	22961	C ₁₁ H ₁₆	1-phenyl-2-methylbutane	3968-85-2	470.15	317.10	2609.44	2		2.1619 × 10 ⁻⁴	
45 46	22962 22963	C ₁₁ H ₁₆ C ₁₁ H ₁₆	1-phenyl-3-methylbutane	2049-94-7 2049-95-8	472.05 465.53	313.35 326.41	2578.57 2686.04	2		2.1878 × 10 ⁻⁴ 2.1002 × 10 ⁻⁴	
	22964	C ₁₁ H ₁₆	2-phenyl-3-methylbutane	4481-30-5	461.15	335.48	2760.75	2		2.0434 × 10 ⁻⁴	
48	22965	C ₁₁ H ₁₆	1-phenyl-2,2-dimethylpropane	1007-26-7	459.15	339.71	2795.55	2	11.21	2.0180 × 10 ⁻⁴	-
49	23006	C ₁₁ H ₁₆	1,2,4-trimethyl-6-ethylbenzene		486.15	286.86	2360.62	2	13.28		-
50 51	23007 23008	C ₁₁ H ₁₆ C ₁₁ H ₁₆	1,3,5-trimethyl-2-ethylbenzene pentamethylbenzene	3982-67-0 700-12-9	485.55 504.55	303.80 255.64	2500.00 2103.68	1		2.2565 × 10 ⁻⁴ 2.6816 × 10 ⁻⁴	
	25025	C ₁₂ H ₁₈	hexylbenzene	1077-16-3	499.26	264.25	2380.29	2		2.3700 × 10 ⁻⁴	-
53	25026	C ₁₂ H ₁₈	1,4-dipropylbenzene	4815-57-0	481.77	294.85	2655.92	2	11.80	$2.1240 imes 10^{-4}$	1,2
	25027	C ₁₂ H ₁₈		25321-09-9		310.32	2795.28	2		2.0182 × 10 ⁻⁴	
55 56	25032 25033	C ₁₂ H ₁₈ C ₁₂ H ₁₈	1,2,3-triethylbenzene 1,2,4-triethylbenzene	42205-08-3 877-44-1	490.66 491.15	278.87 278.02	2512.01 2504.32	2	12.48 12.51	2.2457 × 10 ⁻⁴ 2.2526 × 10 ⁻⁴	-
57	25034	C ₁₂ H ₁₈	1,3,5-triethylbenzene	102-25-0	489.05	281.70	2537.47	2		2.2232×10^{-4}	
58	25043	C ₁₂ H ₁₈	1,2,4-trimethyl-5-isopropylbenzene	10222-95-4	494.15	272.84	2457.70	2		2.2954 × 10 ⁻⁴	
59	25044	C ₁₂ H ₁₈	4-tert-butyl- <i>o</i> -xylene	7397-06-0	478.15	301.60	2716.75	2		2.0765 × 10 ⁻⁴	
60 61	25046 25049.1	C ₁₂ H ₁₈ C ₁₂ H ₁₈	hexamethylbenzene 2,4,6-trimethyl-1-propylbenzene	87-85-4	536.60 494.15	209.15 298.24	1883.93 2686.50	2	16.64 11.67	2.9944 × 10 ⁻⁴ 2.0999 × 10 ⁻⁴	
62	26983	C ₁₃ H ₂₀	heptylbenzene	1078-71-3	519.25	233.15	2281.72	2		2.4724 × 10 ⁻⁴	-
63	26984	C ₁₃ H ₂₀	1-methyl-2,4-diisopropylbenzene	1460-98-6	497.15	267.76	2620.44	2	11.96	2.1528×10^{-4}	1,2
	000/5	C ₁₄ H ₂₂	octylbenzene	2189-60-8	537.55	207.90	2196.51	2	14.27	2.5683 × 10 ⁻⁴	1,2
64 65	28365 28366	C ₁₄ H ₂₂	1,2,3,4-tetraethylbenzene	642-32-0	524.16	226.09	2388.66	2	13.12	2.3617 × 10 ⁻⁴	1.0

			TABLE 1. SOLUBILITY OF W	ATER IN BE	NZENE	DERIVA	TIVES (C	ontinu	ued)		
						ty of Wate er million			-	w Constant (F	1)
No.	ID	FORMULA	NAME	CAS No.	Т _в (К)	\$ @25°C, ppm (wt)	S @25°C, ppm (mol)	code	H @ 25°C, atm/mol frac	H @ 25°C, atm/mol/m ³	code
66	28367	C ₁₄ H ₂₂	1,2,3,5-tetraethylbenzene	38842-05-6	522.00	229.17	2421.20	2	12.94	2.3300×10^{-4}	1,2
67	28368	C ₁₄ H ₂₂	1,2,4,5-tetraethylbenzene	635-81-4	523.16	227.51	2403.67	2	13.04	2.3470×10^{-4}	1,2
68	28369	C ₁₄ H ₂₂	1,4-di- <i>tert</i> -butylbenzene	1012-72-2	510.43	246.39	2603.16	2	12.04	2.1671 × 10 ⁻⁴	1,2
69	29455	C ₁₅ H ₂₄	nonylbenzene	1081-77-2	555.20	186.15	2111.58	2	14.84	2.6716×10^{-4}	1,2
70	29456	C ₁₅ H ₂₄	1,2,4-triisopropylbenzene	948-32-3	517.15	236.24	2679.81	2	11.70	2.1051 × 10 ⁻⁴	1,2
71	29457	C ₁₅ H ₂₄	1,3,5-triisopropylbenzene	717-74-8	511.15	245.29	2782.43	2	11.26	2.0275 × 10 ⁻⁴	1,2
72	29458	C ₁₅ H ₂₄	3,5-di- <i>tert</i> -butyltoluene	15181-11-0	517.15	236.24	2679.81	2	11.70	2.1051 × 10 ⁻⁴	1,2
73	30357	C ₁₆ H ₂₆	decylbenzene	104-72-3	571.04	168.57	2043.41	2	15.34	2.7607 × 10 ⁻⁴	1,2
74	30358	C ₁₆ H ₂₆	pentaethylbenzene	605-01-6	550.16	192.12	2328.89	2	13.46	2.4223×10^{-4}	1,2
75	30361	C ₁₆ H ₂₆	(1-methylnonyl)benzene	4537-13-7	562.65	177.66	2153.65	2	14.55	2.6194×10^{-4}	1,2
76	30361.1	C ₁₆ H ₂₆	(2,4,6-trimethylheptane)benzene		562.65	202.52	2455.00	1	12.77	2.2979 × 10 ⁻⁴	1,2
77	31034	C ₁₇ H ₂₈	undecylbenzene	6742-54-7	586.40	153.11	1975.20	2	15.87	2.8561 × 10 ⁻⁴	1,2
78	31730	C ₁₈ H ₃₀	dodecylbenzene	123-01-3	600.76	139.94	1914.27	2	16.37	2.9470 × 10 ⁻⁴	1,2
79	31731	C ₁₈ H ₃₀	hexaethylbenzene	604-88-6	571.16	168.44	2304.17	2	13.60	2.4483×10^{-4}	1,2
80	31732	C ₁₈ H ₃₀	1,2,4,5-tetraisopropylbenzene	635-11-0	532.15	215.06	2941.86	2	10.65	1.9176 × 10 ⁻⁴	1,2
Coc	le: 1 = do	ata, 2 = esti	imate T _B — boiling point tempe	erature, K							

Example 1. In hydrocarbon processing, toluene (C₇H₈) comes into contact with water at ambient conditions (25°C, 1 atm). Organic and aqueous phases are separated. Estimate the concentration of water at saturation in the toluene after separation.

Substituting coefficients and boiling points into Equation (1) yields:

 $\log_{10}(S) = 3.780 - 2.720 \times 10^{-3} \times 383.78$ = 2.73612

$$S = 544.65 \text{ ppm (wt.)}$$

Example 2. Consider a toluene (C_7H_8) spill into a body of water at ambient conditions (25°C, 1 atm). After separation, the concentration of water in the toluene at the surface is 0.00277 mole fraction. Estimate the concentration of water in the vapor at the surface.

From thermodynamics at low pressure, the vapor concentration is given by: $y = H / (P_{tot} \times X)$

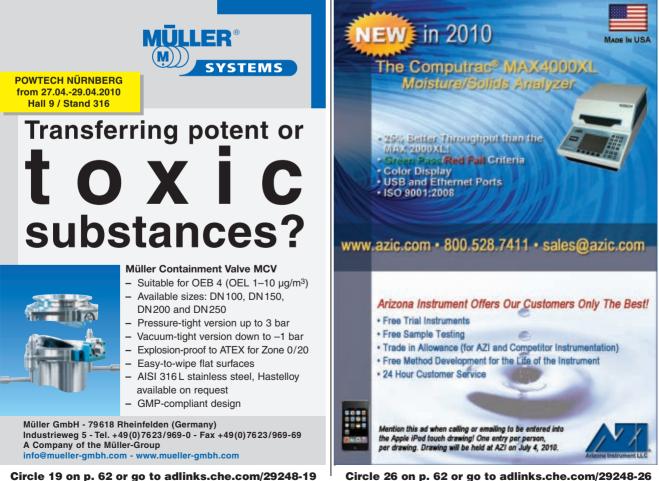
where:

H = Henry's Law constant

X =mole fraction

 $P_{tot} = \text{total pressure}$

Substitution of Henry's Law constant from the table, total pressure $(P_{tot} = 1 \text{ atm})$ and liquid concentration



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into the above equation yields: $y = 11.31 / (1 \times 0.00277) = 0.0313$ y = 3.13 mol %

Edited by Scott Jenkins

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Would You Use a Safety PLC for Process Control?

Ensure unambiguous independence of the control and safety layers of protection

Robin McCrea-Steele Invensys Operations Management

ith today's state-of-the-art digital technology, the sky seems to be the limit for doing everything with one system platform. However, with the exception of a few very specific applications, using a safety PLC (programmable logic controller) for process control is not a good choice. Safety PLCs are designed and optimized to operate in a *static* environment, where action is only taken when a process variable approaches an unsafe region and cannot be controlled by a process control system, such as a distributed control system (DCS). Similarly, in the reverse situation, a DCS should not be used for ESD (emergency shutdown) safety applications.

It is very important to understand that the design requirements for a safety PLC operating in a static, vigilant mode rely on a very high level of online auto-diagnostics while operating in a fail-safe manner. The internal auto-diagnostic routines are continually toggling the discrete values to assure that the devices will operate when a process demand requires action.

Conversely, control systems are designed and optimized to operate in a *dynamic* environment, in which measurements and actions are continuous and operators and engineers have less rigorous constraints on access for tweaking and changing settings.

Internal failures in control systems are usually overt, as the dynamics of

FIGURE 1. The layers of protection for a chemical plant, including both control and safety, should be unambiguously independent

the mode of operation tend to reveal the failures to plant operators. Internal failures in safety systems, on the other hand, may be overt but also can be covert and dangerous, because they are masked due to the static mode of operation. If the internal auto-diagnostics of the safety system cannot detect a dangerous failure, it will be revealed either by online external proof testing or by a process demand. The latter is the critical issue, because revealing a dangerous covert failure by a process demand mostly realizes the hazard being protected against, with consequent release of energy that may result in fires, explosions, injuries, loss of life and other safety incidents.

As can be derived from the above discussion, the design requirements for a safety system and a process control system are completely opposing and conflicting. Chemical engineers responsible for operating potentially hazardous process plants should be aware of these differences when encountering vendors that have not been traditional players in the safety arena, but now design safety systems based on their existing DCS platforms. Think of your DCS as a Ferrari Testarossa with a 12-cylinder engine optimized to perform at 200 mph and designed with the goal of crossing the finish line first. Now think of your safety system as a Volvo optimized to perform best at 65 mph and designed with all the airbags and safety features for secure transportation. With this in mind, you would not likely think of driving your kids to school in a modified car based on a design optimized to run at incredibly high speeds revving at 7,500 rpm, nor would you dream of racing in a "juiced-up" Volvo sedan.

Community emergency response

Plant emergency response

Physical protection (containment) Physical protection (relief devices)

Automatic SIS Critical alarms and manual intervention

Basic control

Basic design

In this article, we discuss the advantages and disadvantages of using a common platform approach for safety and control, while adhering to the spirit of IEC 61511/ANSI ISA S84.00.01 standards and good engineering practices in the chemical process industries (CPI).

Independent protection layers

The basis for the concept of defense in depth and diversity (D3) and independent protection layers (IPL) at the heart of all the international safety standards (including IEC 61508 and

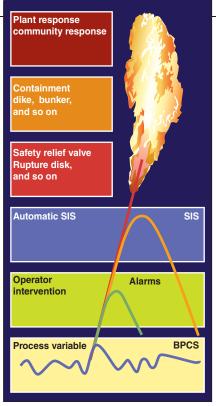


FIGURE 2. Each independent protection layer (IPL) is designed to independently protect against specific hazards. Shown here are the specific characteristics of true layer and action independence

ANSI ISA 84.00.01-2004 [IEC 61511 Mod]), is that every layer of protection, as shown in Figure 1, including both control and safety, should be unambiguously independent. Some of the reasons for this basic requirement are to avoid common cause faults, minimize systematic errors and provide security against unintentional access, sabotage and cyber-attacks.

If two IPLs are designed based on the same hardware-software platform, the effect of common-cause and systematic errors is a merge of two layers of protection, leading to a safety incident waiting to happen. It is not enough to separate the safety functions in a functionally separate module if the hardware and software are based on the same DCS platform.

Regarding the cyber-security risks, if the DCS and SIS (safety instrumented system) are sharing communication networks, as in network traces on DCS-controller backplane-embedded systems, the amount of risk that the SIS is exposed to by the outside world, network is heightened by the number of viruses or other maladies that the DCS network might encounter.

On the other hand, with a physically

AICHE-CCPS "GUIDELINES FOR SAFE AND RELIABLE PROTECTIVE SYSTEMS" SECTION C

C.1 Independence. For a protection layer to be considered independent, its performance should not be affected by the occurrence of the initiating cause, its consequences, or by the failure of another protective function used to reduce the risk of the same hazardous event. The correct operation of the protection layer should not be conditional on any other layer, and its separation from other layers should be unambiguous.

C.2 Functionality. The protection layer must be capable of responding effectively within the time required by stopping the propagation of the initiating cause, even in the presence of other protection layer failures. This requirement along with the core attribute of independence generally results in the use of separate equipment and management systems for each protection layer.*

The reduction of the system to its individual functions allows function classification and provides traceability between the design and management of the function and the required risk reduction.

*Emphasis by author

separate and diverse safety system, there is inherent, built-in protection from these risks.

Each IPL is designed to independently protect against the hazard for which it has been designed to safeguard. Figure 2 summarizes the key factors in effective IPLs. From a process safety point of view, one of the duties of the DCS is to reduce the number of demands on the SIS. A demand on the SIS implies that the control system has failed to keep the process within the safety margin and the process is now relying on the SIS to protect against the hazard.

In essence, with truly, unambiguously independent protection layers, the potential hazard will occur "only when both the BPCS (basic process control system) and the SIS fail". However, if the IPLs are not truly independent, systematic errors and common-cause faults could result in both the DCS and SIS failing simultaneously, which could lead to a safety incident.

Interpreting safety standards

IEC 61511-1 clause 11.2.4 states that the BPCS shall be designed to be separate and independent to the extent that the functional integrity of the SIS is not compromised. The caution here is that these are minimum benchmark requirements and may not provide adequate risk reduction in many CPI applications. Still, several automation vendors have interpreted the above clause 11.2.4 to mean that the standard does not require physical separation or diversity. Such a conclusion is dangerous.

However, another section of the same standard IEC 61511-1, clause

9.5, addresses the requirements for preventing common-cause, commonmode and dependent failures. Clause 9.5.2 states that the assessment shall consider (a) independency between protection layers, (b) diversity between protection layers, (c) physical separation between protection layers and (d) common-cause failures between protection layers and the BPCS.

The question is how to conform to clause 11.2.4 without physical and diverse separation. Systematic, common-cause and software errors form an integral component of the overall safety assessment.

The recently published AIChE Center for Chemical Process Safety (CCPS) book, titled "Guidelines for Safe and Reliable Protective Systems" cautions that the international safety standards are performance benchmarks for minimum requirements. It further defines in Section C, p. 301 that "independence, functionality, integrity, reliability, auditability, access security and management of change are fundamental characteristics of an independent protection layer (IPL)." This section, shown in the Box above, explains why a safety logic solver that is embedded within the same platform as the control system, even if it using separate modules, does not meet the requirements of an independent layer of protection.

Is TÜV certification enough?

International safety-application standards require that manufacturers document compliance of SIS logic solvers to IEC61508. Some people may think that a third-party certificate of equipment compliance based on such standards is sufficient. A TÜV certificate

Engineering Practice

of compliance goes a long way, and although essential, it should not be the only criterion. The ultimate responsibility for documentation lies with the plant operating company's management, not the vendor.

A third-party certificate of compliance for the SIS logic solver will validate the design and fail-safe suitability for use in a safety instrumented function up to the SIL (safety integrity level) claim limit. For systems where the SIS logic solver hardware and software is based on the same platform as the control system (DCS), the certification will validate the fact that failures in the DCS will not affect the SIS safety functions.

The first deficiency is that the certification does nothing to avoid the common-cause failures of the SIS and DCS, which are based on the same hardware-software platform. Nor does the certification say anything about the systematic errors inherent in using the same platform for SIS and DCS. The certification basically validates the "functional separation" and non-interference of control-system failures on the SIS, firewalls and password-based access protection.

Independence of the layers of protection in the plant is also not part of the SIS logic-solver certificate. Rather, this determination is a responsibility of the operating plant company.

Compliance to the "functional separation" requirements of IEC61511 is enough to obtain a TÜV certificate. However, an independent layer of protection needs to be "unambiguously" independent. If a common-cause error can affect both the DCS and the SIS, then no credit can be taken for the control system as an independent layer of protection.

Therefore, although a TUV certificate for a certain SIL-capability limit for the SIS logic solver validates the use of a functionally separate, but common-platform DCS-SIS, great caution needs to be taken in the overall implementation of plant risk-reduction requirements.

A recent study by a major petroleumrefining and energy corporation determined in its review of a TÜV-certified, DCS-embedded safety system (which used separate modules), that although

AICHE-CCPS "GUIDELINES FOR SAFE AND RELIABLE PROTECTIVE SYSTEMS" SECTION F

Section F.2.3: Future Technology

Most owner/operators continue the practice of implementing separate, and often diverse, platforms for the BPCS and SIS*, following the well-proven, defense in depth strategy that supports both safety and reliability. With a physically separate BPCS controller and SIS logic solver, independence is easier to assess and manage over the process equipment lifetime. Independence allows the owner/operator to implement different management systems for the BPCS and the SIS; the BPCS management system may be more flexible and less rigorous than the SIS management systems.

Section F.3.5: Logic Solver Separation

The interaction between the BPCS and SIS is now much more complex. Field devices are often shared as discussed in Section F.4, and there may be extensive communication between the systems as discussed in Section F.6. However, experienced engineers and many good engineering practices continue to recommend implementing the SIS in a physically separate logic solver from the control functions.

A major justification for separation is reduced long-term administrative costs. When layers are combined, the management systems of the highest layer applies. Means should be provided to restrict access, to limit communication to other systems and to control system changes. Generally the cost of separation is significantly less than the administrative cost to maintain the required rigor. The administrative rigor must be maintained for the life of the system, including the provision for necessary resources to verify and audit compliance.

Adequate separation is achieved by administrative controls and physical means. Physical separation is provided at the system level by executing the functions in separate and, often diverse logic solvers. Access security and management of change is enhanced by physically separate systems. When the BPCS is physically separate from the SIS, the need to access the SIS is reduced and the BPCS can be managed under a less rigorous management system.

Separation ensures that the BPCS and SIS are not dependent on each other to operate. It also provides a clear and unambiguous distinction between the BPCS and SIS, which supports long-term access security and management of change. Separation also ensures that when maintenance and testing is conducted on one system the other remains available.

*Emphasis by author

non-interfering, the BPCS-SIS separation could not be adequately satisfied as an IPL. This operating company concluded that, because common communication traces were used by both the BPCS and the SIS equipment on the same carrier, no credit could be gained for the BPCS as an independent layer of protection.

Common-platform approach

Certainly, a "common-platform" approach, using similar hardware and software dedicated for control and safety functions, respectively, can arguably provide the potential for some cost savings. However, it is widely acknowledged that utilizing separate, independent, and diverse hardware and software for process safety and control is the optimal way to protect against potentially catastrophic common-cause and systematic design and application errors.

Some very specific applications such as surge control, turbomachinery speed control or burner management systems are examples of the exceptions. In these cases, it is recommended to implement the applications in a fault-tolerant safety system, rather than in a DCS.

Different vendors offer varied degrees of integration and solutions. The question is how to provide an integrated control and safety solution with advanced functionality and productivity without compromising safety and security.

Tight integration

There is undoubtedly a very good case to be made for tight integration of control and safety from an operationsand-productivity point of view. Some of the major potential benefits include:

- Seamless integration
- Time synchronization
- Elimination of data mapping duplication
- Common HMI (human machine interface)
- Compatible configuration tools
- Minimized set of spare parts
- Single operator and maintenance training requirements
- All of the above are great benefits for

productivity and maintenance. However, merging control and safety too closely could negate these advantages. Therefore, users need to consider the side effects of using a common platform. How is the integrity of each independent protection layer guaranteed? Could a loss of communication cause the SIS controller to fail to function? Can a common network expose the SIS to security risks? Does a DCSembedded safety-logic solver pose concerns of side effects and hidden costs?

Potential benefits can turn into liabilities if they come at the expense of safety and security, and most times they can even increase lifecycle costs.

Smart integration

The AIChE-CCPS "Guidelines for Safe and Reliable Protective Systems" discusses future technology and logic solver separation in Section F (Box, p. 46). The conclusion is that integrating physically separate and diverse unambiguously independent safety and control systems - with interoperability at the information, configuration, asset management and HMI levels is the safest, most reliable and lowest lifecvcle cost solution. It must also be noted that all the capabilities of field diagnostics and asset management, including partial stroke testing, can be implemented effectively through interoperable integration.

Integrating smartly at an interoperability level requires additional measures to ensure that the safety of the communications and control commands are policed by the safety system.

When implementing an automation infrastructure for a potentially hazardous process, consider defense in depth and diversity, integrating safety and control smartly with an interoperable solution.

Final analysis

Chemical engineers responsible for operating potentially hazardous process plants should evaluate the criticality of the application, and determine if it is appropriate to use a process safety system that is designed based on the same hardware-software platform as the DCS.

The assessment should consider the downsides of using two layers of pro-

tection based on the same platform, where the design requirements are completely opposing and conflicting and where the implications of common-cause and systematic errors are conducive to higher operational risks.

To ensure that process safety risks are minimized and reliability is optimized, it is important to implement control and safety utilizing the appropriate tools specifically designed for each job.

- For process *control*, use a DCS or other control system designed for a dynamic operations environment
- For process *safety*, use a fault-tolerant safety PLC designed for a static operations environment, maximizing auto-diagnostics and proof testing
- Use physically and functionally separate and diverse systems for safety and control IPLs
- Use interoperable integration of safety and control systems at the information, configuration, asset man-

- agement, HMI and enterprise levels
- Do not cut corners by using a DCSembedded safety system

A plant can achieve maximum economic benefit by smartly integrating separate and diverse control and safety systems at the interoperability level, without compromising safety and security.

Edited by Gerald Ondrey

Author



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Rheinland Functional Safety Program. He is an AIChE Member, ISA Senior Member and SP84 committee member working on the Safety Fieldbus task force. McCrea-Steele is a safety consultant to the FF-SIS Rollout management team. He is a patent holder for continuous online safety integrity monitoring methodology, and he specializes in process safety consulting and risk assessments.



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Plugging is not an issue with this phosphorus analyzer

The ChemScan mini oP analyzer (photo) for *ortho*-phosphorus analysis features flow paths designed to avoid plugging. The analyzer uses large orifice sizes, compared to capillary tubes and needle valves of most other analyzers. The unit operates for three months between reagent addition, and any of the analyzer's components can be replaced within 5 min. — ASA Analytics, Waukesha, Wisc. www.chemscan.com

Deposits are not a problem for this process refractometer

To meet the requirements of the chemical industry, versions of the PIOX R400 process refractometer (photo) for use in explosive atmospheres from ATEX Zones 2 to 0 are now available. The device measures the light transmitted through the entire volume of the sample, instead of a thin film between prisms. As a result, higher resolution and extremely high precision are achieved. Also, the measurement is less sensitive to deposits forming on the prism compared to criticalangle reflectometry. The instrument uses dual beams of light, which allows for completely drift-free differential measurements, even with deposits. ---Flexim GmbH, Berlin, Germany www.flexim.de

This gas sensor can now be calibrated remotely

Calibrating the Model S4000CH Combustible Gas Detector with a remotely installed sensor is now easier than ever with the new Automatic Remote Gas Calibrator (ARGC; photo). The ARGC, which consists of a Remote Gas Calibrator and a solenoid valve, blocks ambient air and redirects the calibration gas to the catalytic bead sensor for calibration or testing sensor accuracy. The instrument can calibrate gases at wind velocities as high as 50 mph and maximum temperatures of 75°C. The S4000CH is a hydrocarbon gas detector suitable for use in SIL 3 applications. — General Monitors, Lake Forest, Calif.



Electro-Chemical Devices

Measure fluoride levels

over a wide concentration range Accurate fluoride-ion measurements are required for applications including semiconductor fabrication and glass manufacturing. The S10 and S17 Fluoride pIon Sensors (photo) measure the activity of free fluoride ions in solution in concentration from 0.02 to 2,000 ppm over a pH range from 5-8. They operate over a temperature range of 0 to 80°C. The sensors can be integrated with the company's C22 Analyzer to provide automatic pH compensation. — Electro-Chemical Devices. Inc., Irvine, Calif. www.ecdi.com

Measure dissolved O₂ with this amperometric system

This firm offers a comprehensive range of amperometric systems for measuring dissolved oxygen, with sensors for wide application coverage, retractible housings for sensor extraction without process interruption, and transmitters for signal conversion and connection to process control systems using HART, Foundation fieldbus or Profibus. The complete measurement system is available with ATEX Zone 1/FM Class 1 Div. 1 approval, if required. — Mettler-Toledo, Inc., Columbus, Ohio www.mt.com

This phosphate analyzer consumes less reagents

The new Navigator 600 phosphate analyzer (photo) greatly reduces the amount of required reagents (1/10th that of competitive units), and maintenance, thereby substantially reducing the cost of phosphate monitoring. Aimed at phosphate-dosed, highpressure boilers, the unit provides accurate monitoring over the concentration range of 0 to 15 ppm of PO_4^{-3} . The analyzer is available in single- or multi-stream configurations, enabling up to six streams to be monitored sequentially. It provides current loops, Ethernet and optional Profibus DP V1 outputs. — ABB Instrumentation, Warminster. Pa.

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Utilizing a high-performance, lownoise flame-ionization-detector (FID) subsystem and logarithmic amplifier that decreases drift and thermal noise, the Servopro FID (photo) offers accurate (100 ppb resolution) measurement of hydrocarbons. With all flow electronically regulated and temperature regulated, the system offers a "Lock Range" facility to allow the operator to fix the unit within a desired range, as well as an automatic ranging feature that changes the measurement parameters according to impurities detected. - Servomex Group Ltd., Crowborough, U.K.

www.servomex.com

This gas analyzer has a two-laser platform

Two separate lasers are installed in the Model 5100HD gas analyzer (photo). They share a common optical path through the sample, allowing the instrument to measure two analytes simultaneously, which results in significant cost savings. The system is based on TDLAS (tunable diode-laser absorption spectroscopy) and can measure a combination of any two of the following: water vapor, CH_4 , H_2S , CO and O_2 in process or fluegas. The instrument is designed to handle sample gases with high dew points. The standard version has its sample cell heated to 100°C, and another version is available for 150°C. — Ametek Process Instrumentation, Pittsburgh, Pa. www.ametekpi.com

A photometer that can store 16 parameters

The new version of the Photometer P15 plus (photo) has a measured-value memory card and a realtime clock. It can store the values of up to 16 of the most used water-treatment quality parameters, together with their times of measurement. Factors that could impair measurement — such as excess turbidity of the water sample — are automatically detected and displayed. Parameters such as pH, acid capacity, free and total chlorine, chlorine dioxide, bromine, ozone and cvanuric acid can be measured quickly, easily and precisely. - Siemens Water Technologies, Warrendale, Pa. www.siemens.com

www.siemens.com

Web access is a feature of this gas analyzer

The Rosemount Analytical X-Stream XE process gas analyzer (photo) combines Web-browser-based accessibility with advanced processing capabilities. The ability to remotely manage the analyzer greatly simplifies diagnostics, speeds the troubleshooting process, and reduces the number of trips into the field by an estimated 50%. The instrument offers a programmable logic controller that enables automation of the sample-handling system and online programming through the Web browser. An enhanced data logger offers improved data tracking, including a Namur status indicator, improved event logging and a new "cal log" file. — Emerson Process Management, Houston, Tex.

www.emersonprocess.com

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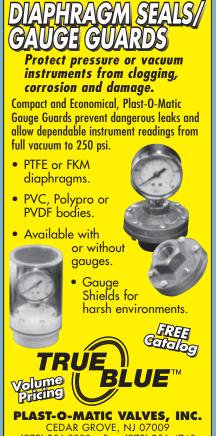
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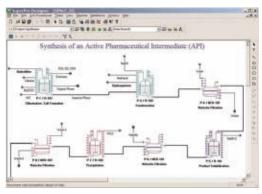
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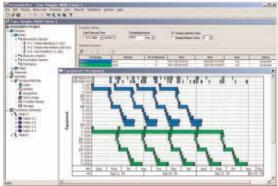
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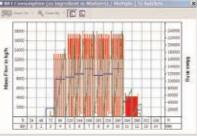
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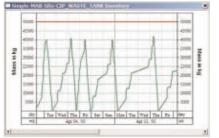
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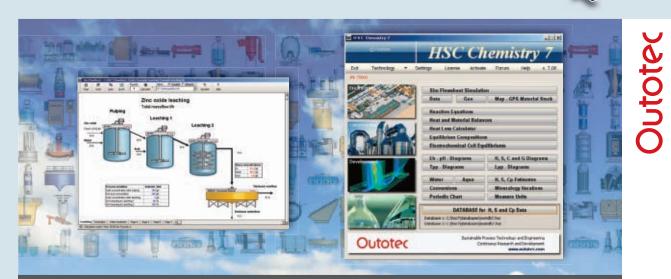
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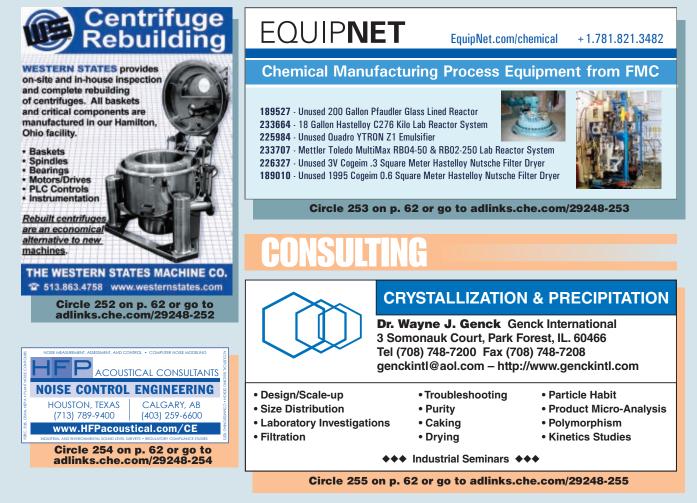
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KBR to provide licensing of its Purifer Ammonia technology

February 19, 2010 — KBR (Houston; www.kbr. com) has been awarded a contract by Matix Fertilisers and Chemicals Ltd. (MFCL) — a Matix Group Co. owned by the Kanoria family in India — to provide licensing and engineering services for MFCL's grassroots 2,200 metric tons per day (m.t./d) ammonia plant located in West Bengal, India. KBR will license its Purifier Ammonia technology for the Matix plant, which will be based on coal-bed methane. The use of coal-bed methane as feedstock in the ammonia synthesis process is a relatively new application that provides an alternative to the use of natural gas in areas where coal is abundant.

Pöyry awarded board-mill engineering for SAICA

February 2, 2010 — Pöyry Plc. (Helsinki; www. poyry.com) has been awarded an engineering contract by SAICA Containerboard UK Ltd. for one of the world's most advanced recycled paper mills to be built at Partington Wharfside, near Manchester, U.K. The new paper mill will produce 425,000 ton/yr of high-performance lightweight fluting and testliner grades to be used in the manufacture of corrugated boxes. The 100% recycled, fiber-based mill is scheduled to start up at the beginning of 2012.

BMS opens world's largest CNT pilot facility

February 1, 2010 — Bayer MaterialScience (BMS; Leverkusen, Germany; www.bayermaterialscience.com) has opened a new pilot facility for the manufacture of carbon nanotubes (CNTs) at Chempark Leverkusen. The company has invested some €22 million in the planning, development and construction of the facility, which is the largest of its kind in the world and has a capacity of 200 m.t./yr.

MERGERS AND ACQUISITIONS

Abbott completes acquisition of Solvay Pharmaceuticals

February 16, 2010 — Abbott (Abbott Park, III.; www.abbott.com) has completed its €4.5 billion acquisition of Belgium-based Solvay Pharmaceuticals. Solvay Pharmaceuticals is now part of Abbott's global Pharmaceutical Products Group. Werner Cautreels, chief executive officer of Solvay Pharmaceuticals, will serve in a transitional role and will then leave the company. The transaction also includes payments of up to €300 million if certain sales milestones are met between 2011 and 2013.

Mitsubishi Chemical and Pioneer form alliance on OLED lighting

February 9, 2010 – Mitsubishi Chemical Corp. (Tokyo, www.m-kagaku.co.jp) and Pioneer Corp. (Kanagawa, both Japan; www. pioneer.jp/index-e.html) have entered into an alliance on their organic light-emitting diode (OLED) lighting business as well as a capital alliance strengthening their company relations. Mitsubishi Chemical is aiming to begin full-scale mass production and launch of illumination appliances in 2011. With this business move, Mitsubishi Chemical will use OLED lighting panels supplied by Pioneer. It is also looking into performing joint research on printable OLED lighting development, as well as their commercialization. Both companies are currently performing joint research on OLED lighting panels that use printable hole injecting material (HIM) and new emitting materials. MItsubishi Chemicals is looking into the commercialization of printable OLED lighing.

INEOS Group to sell fluorochemicals business to Mexichem Fluor S.A

February 2, 2010 - The Ineos Group (Lyndhurst, U.K.; www.ineos.com) has agreed to sell its fluorochemicals business to Mexichem, a Latin American producer of PVC pipes and resin, chloralkali, hydrofluoric acid and fluorspar. The deal comprises the international business and assets related to INEOS' fluorochemical operations located in North America, Europe, and Asia. It is expected that on completion of the transaction, the business will become an integrated, global producer of speciality fluorochemicals with annual sales revenue of over \$500 million. The sale is expected to be completed at the end of March, subject to necessary regulatory filings and approvals, including bank consent and approvals under applicable antitrust laws and regulations.

Total Safety acquires ICU Environmental, Health & Safety

February 1, 2010 — Total Safety (Houston; www.totalsafety.com) has acquired ICU Environmental, Health & Safety, which provides services related to industrial hygiene, safety inspections and audits, process safety management services, regulatory compliance, environmental air-permitting consulting, risk assessment and others. Specific financial terms of the acquisition were not disclosed.

Brain and Bayer Schering Pharma cooperation

February 2, 2010 - Brain AG (Zwingenberg; www.brain-biotech.de) and Bayer Schering Pharma AG (Berakamen, both Germany; www.bayerscheringpharma.de) are cooperating in the field of production process optimization of steroid compounds. The goal of the collaboration is the energyefficient and thus sustainable fermentative production of steroid compounds using optimized microbial production strains, taking plant-derived raw materials as the starting material. With so-called "designer bugs" it is the goal, to achieve a higher yield by a reduced energy input. This will lead to an increase of efficiency in the production process, coupled with a reduction of the output of greenhouse gases.

Braskem acquires Sunoco Chemicals' PP assets

February 1, 2010 - Braskem S.A. (Sao Paolo, Brazil; www.braskem.com.br) has signed an agreement with Sunoco, Inc. (Sunoco), a U.S.-based oil company, for the acquisition by Braskem of the polypropylene (PP) business of Sunoco Chemicals, Inc., based in Philadelphia, Pa. Sunoco will receive \$350 million for the PP-asset-related shares in Sunoco Chemicals. Sunoco Chemicals' polypropylene production capacity is 950,000 ton/yr. Its three plants located at La Porte, Tex., Marcus Hook, Pa., and Neal, W.Va. account for approximately 13% of installed, U.S.-polypropylene-production capacity. In addition to Sunoco's industrial units, the acquisition also includes a technology and development center in Pittsburgh, Pa.

Total and ERG create a joint venture in the Italian petroleum refining business

January 28, 2010 — Total (Courbevoie, France; www.total.com) and ERG (Genoa, Italy; www.erg.it) have signed an agreement to create a joint venture in the Italian marketing and petroleum refining business. Total and ERG will hold equity stakes of 49% and 51%, repectively. Created through the merger of Total Italia and ERG Petroli, the joint venture will be called TotalErg.The shareholder pact calls for joint governance and operating independence for the new entity.The transaction is subject to the approval of competition authorities.

Dorothy Lozowski

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March 2010; VOL. 117; NO. 3

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Economic Indicators

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Dec '08

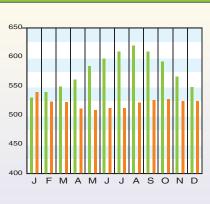
Nov '00

CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

10	57.	50	- 1	00)

(1757-57 = 100)	Prelim.	Final	Final	Annual Index:
CEIndex	524.2	524.0	548.3	2001 = 394.3
Equipment	618.4	618.0	654.3	0000 005 0
Heat exchangers & tanks	554.2	555.9	618.2	2002 = 395.6
Process machinery	597.9	601.0	623.2	2003 = 402.0
Pipe, valves & fittings	776.3	768.2	806.1	2004 = 444.2
Process instruments	417.5	413.9	397.0	
Pumps & compressors	895.2	895.2	891.3	2005 = 468.2
Electrical equipment	467.2	465.9	459.7	2006 = 499.6
Structural supports & misc	620.0	624.2	684.0	
Construction labor	331.2	331.1	328.3	2007 = 525.4
Buildings	494.4	493.7	503.7	2008 = 575.4
Engineering & supervision	343.2	343.8	349.9	

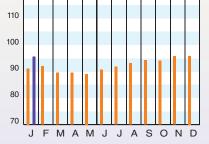
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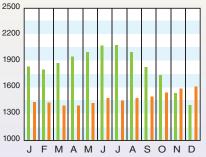
Starting with the April 2007 Final numbers, several of the data series for labor and compressors have been converted to accommodate series IDs that were discontinued by the U.S. Bureau of Labor Statistics

LAT	EST				PREVIC	ous			YEAR	AGO	b
Jan.'10	=	96.5	Dec.'09	=	95.9	Nov. '09	=	95.8	Jan.'09	=	
Dec.'09	=	1,612.4	Nov. '09	=	1,587.0	Oct.'09	=	1,540.6	Dec.'08	=	1,
Jan.'10	=	71.6	Dec.'09	=	71.0	Nov. '09	=	70.8	Jan.'09	=	
Jan.'10	=	260.1	Dec.'09	=	254.9	Nov. '09	=	251.3	Jan.'09	=	
Jan.'10	=	99.4	Dec.'09	=	98.5	Nov. '09	=	98.5	Jan.'09	=	
Jan.'10	=	150.4	Dec.'09	=	150.9	Nov. '09	=	150.4	Jan.'09	=	
Jan.'10	=	135.1	Dec.'09	=	135.3	Nov. '09	=	137.0	Jan.'09	=	
	Jan.'10 Dec.'09 Jan.'10 Jan.'10 Jan.'10 Jan.'10	Jan.'10 =	Dec.'09 = 1,612.4 Jan.'10 = 71.6 Jan.'10 = 260.1 Jan.'10 = 99.4 Jan.'10 = 150.4	Jan.'10 = 96.5 Dec.'09 Dec.'09 = 1,612.4 Nov.'09 Jan.'10 = 71.6 Dec.'09 Jan.'10 = 260.1 Dec.'09 Jan.'10 = 99.4 Dec.'09 Jan.'10 = 150.4 Dec.'09	Jan.'10 = 96.5 Dec.'09 = Dec.'09 = 1,612.4 Nov.'09 = Jan.'10 = 71.6 Dec.'09 = Jan.'10 = 260.1 Dec.'09 = Jan.'10 = 99.4 Dec.'09 = Jan.'10 = 150.4 Dec.'09 =	Jan.'10 = 96.5 Dec.'09 = 95.9 Dec.'09 = 1,612.4 Nov.'09 = 1,687.0 Jan.'10 = 71.6 Dec.'09 = 71.0 Jan.'10 = 260.1 Dec.'09 = 254.9 Jan.'10 = 99.4 Dec.'09 = 98.5 Jan.'10 = 150.4 Dec.'09 = 150.9	Jan.'10 = 96.5 Dec.'09 = 95.9 Nov.'09 Dec.'09 = 1,612.4 Nov.'09 = 1,587.0 Oct.'09 Jan.'10 = 71.6 Dec.'09 = 71.0 Nov.'09 Jan.'10 = 260.1 Dec.'09 = 71.0 Nov.'09 Jan.'10 = 99.4 Dec.'09 = 254.9 Nov.'09 Jan.'10 = 99.4 Dec.'09 = 98.5 Nov.'09 Jan.'10 = 150.4 Dec.'09 = 150.9 Nov.'09	Jan.'10 = 96.5 Dec.'09 = 95.9 Nov.'09 = Dec.'09 = 1,612.4 Nov.'09 = 1,587.0 Oct.'09 = Jan.'10 = 71.6 Dec.'09 = 71.0 Nov.'09 = Jan.'10 = 260.1 Dec.'09 = 254.9 Nov.'09 = Jan.'10 = 99.4 Dec.'09 = 98.5 Nov.'09 = Jan.'10 = 150.4 Dec.'09 = 150.9 Nov.'09 =	Jan.'10 = 96.5 Dec.'09 = 95.9 Nov.'09 = 95.8 Dec.'09 = 1,612.4 Nov.'09 = 1,587.0 Oct.'09 = 1,540.6 Jan.'10 = 71.6 Dec.'09 = 71.0 Nov.'09 = 70.8 Jan.'10 = 260.1 Dec.'09 = 254.9 Nov.'09 = 251.3 Jan.'10 = 99.4 Dec.'09 = 98.5 Nov.'09 = 98.5 Jan.'10 = 150.4 Dec.'09 = 150.9 Nov.'09 = 150.4	Jan.'10 = 96.5 Dec.'09 = 95.9 Nov.'09 = 95.8 Jan.'09 Dec.'09 = 1,612.4 Nov.'09 = 1,587.0 Oct.'09 = 1,540.6 Dec.'08 Jan.'10 = 71.6 Dec.'09 = 71.0 Nov.'09 = 70.8 Jan.'09 Jan.'10 = 260.1 Dec.'09 = 254.9 Nov.'09 = 251.3 Jan.'09 Jan.'10 = 99.4 Dec.'09 = 98.5 Nov.'09 = 98.5 Jan.'09 Jan.'10 = 150.4 Dec.'09 = 150.9 Nov.'09 = 150.4 Jan.'09	Jan.'10 = 96.5 Dec.'09 = 95.9 Nov.'09 = 95.8 Jan.'09 = Dec.'09 = 1,612.4 Nov.'09 = 1,587.0 Oct.'09 = 1,540.6 Dec.'08 = Jan.'10 = 71.6 Dec.'09 = 71.0 Nov.'09 = 70.8 Jan.'09 = Jan.'10 = 260.1 Dec.'09 = 251.3 Jan.'09 = Jan.'10 = 99.4 Dec.'09 = 98.5 Nov.'09 = 98.5 Jan.'09 = Jan.'10 = 150.4 Dec.'09 = 98.5 Nov.'09 = 98.5 Jan.'09 =

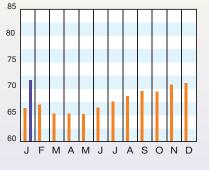
CPI OUTPUT INDEX (2000 = 100) 120



CPI OUTPUT VALUE (\$ BILLIONS)



CPI OPERATING RATE (%)



(1926 = 100)	4th Q	3rd Q	2nd Q	1st Q	4th Q	1500		T	Т
	2009	2009	2009	2009	2008	1485			
M & S INDEX		1,446.4	1,462.9	1,477.7	1,487.2				
Process industries, average		1,515.1	1,534.2	1,553.2	1,561.2	1470			
Cement	1,508.2	1,509.7	1,532.5	1,551.1	1,553.4	1455			
Chemicals	1,483.1	1,485.8	1,504.8	1,523.8	1,533.7	1455			T
Clay products	1,494.3	1,495.8	1,512.9	1,526.4	1,524.4	1440			
Glass	1,400.1	1,400.4	1,420.1	1,439.8	1,448.1				
Paint	1,514.1	1,515.1	1,535.9	1,554.1	1,564.2	1425			
Paper	1,415.8	1,416.3	1,435.6	1,453.3	1,462.9	1410			
Petroleum products	1,617.6	1,625.2	1,643.5	1,663.6	1,668.9	1410			l
Rubber	1,560.5	1,560.7	1,581.1	1,600.3	1,604.6	1395			
Related industries									
Electrical power	1,377.3	1,370.8	1,394.7	1,425.0	1,454.2	1380			
Mining, milling	1,548.1	1,547.6	1,562.9	1,573.0	1,567.5	1365			
Refrigeration	1,769.5	1,767.3	1,789.0	1,807.3	1,818.1	1000			I
Steam power	1,470.8	1,471.4	1,490.8	1,509.3	1,521.9	1350			
	Anni	ual Index:				1335			
2002 = 1,104.2	2004 = 1,178.5	200	6 = 1,302.3	2008	= 1,449.3	1320			1
2003 = 1,123.6	2005 = 1,244.5	200	7 = 1,373.3	2009	= 1,468.6	1	st 2nd	I 3rd Iarter	

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CURRENT TRENDS

All of the major business Aindicators for the CPI continue to climb, although there is a lot of ground to make up before the previous peaks are achieved again.

Meanwhile, the year over year deficit in capital equipment prices (as reflected in the Chemical Engineering Plant Cost Index) is narrowing. December 2009 equipment prices are 4.6% lower than those of the previous December, compared to the widest year over year deficit of 18.9% in July.

Visit www.che.com/pci for more on capital cost trends and methodology.

2009

2008

91.2

66.3

220.6

97.8

145.3

126.0

1,400.5

plant sites & companies

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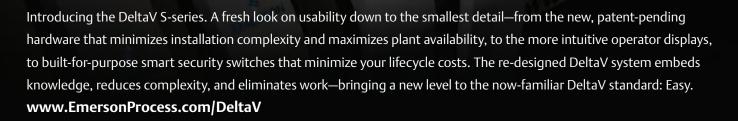
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