CHEMICAL ENGINEERING

February
2012

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Thermal Design of Cooling Towers

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Focus on Spill Response

Membranes for Gas Separation

Oil-injected Screw Compressors

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Particle Size Analysis

Facts at Your Fingertips: pH Electrodes and Temperature Dependence

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Access Intelligence

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TIP-TITE™ Universal Drum Dumper handles drums from 30 to 55 gal (114 to 208 liter)

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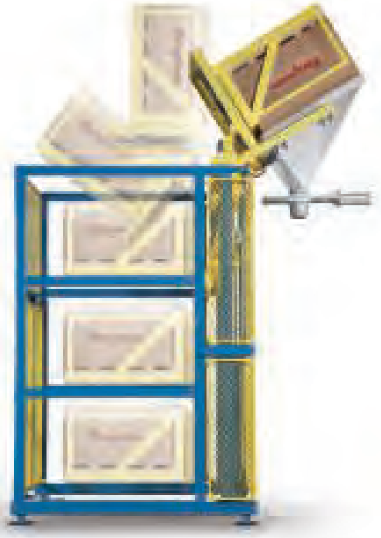

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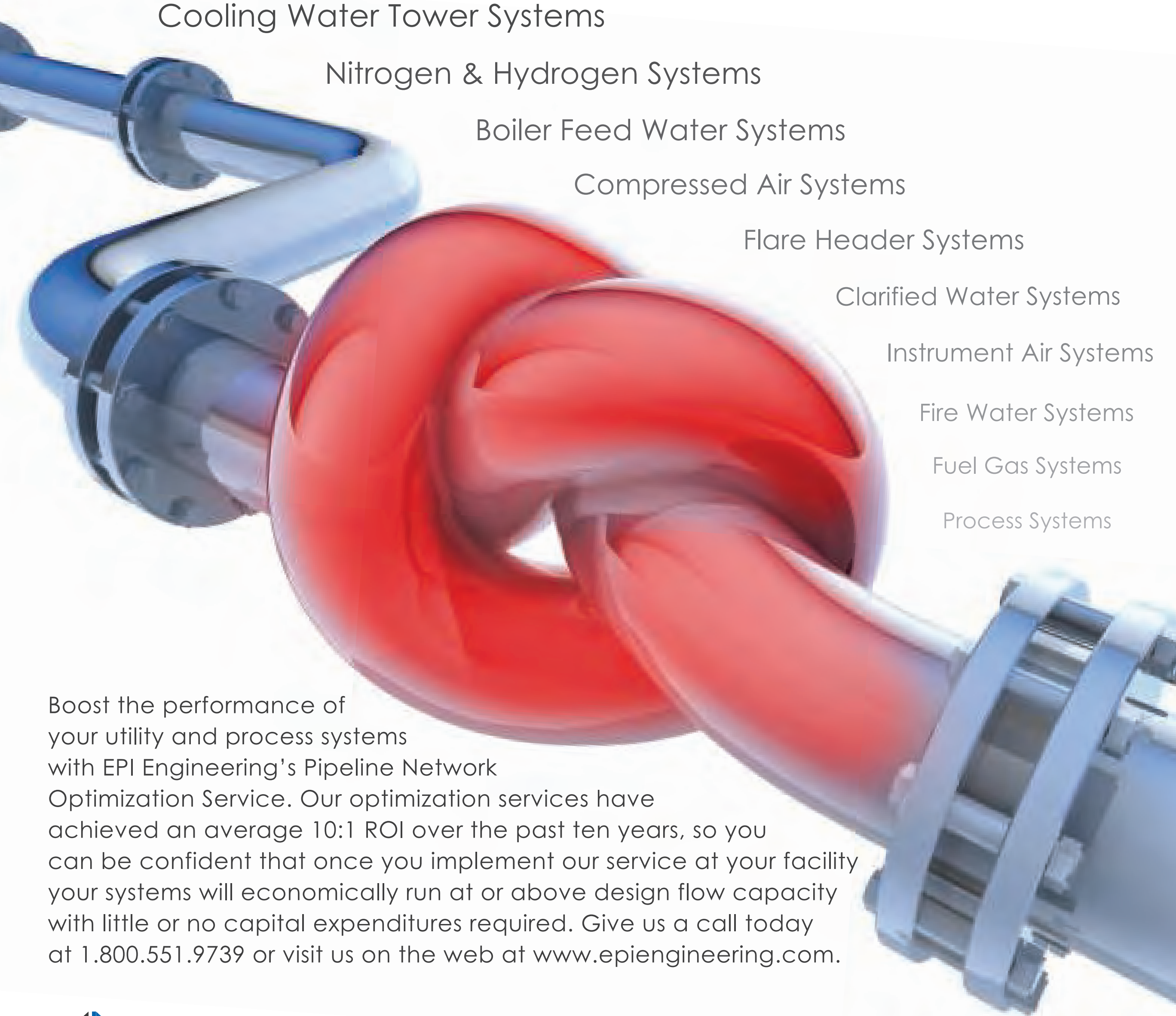

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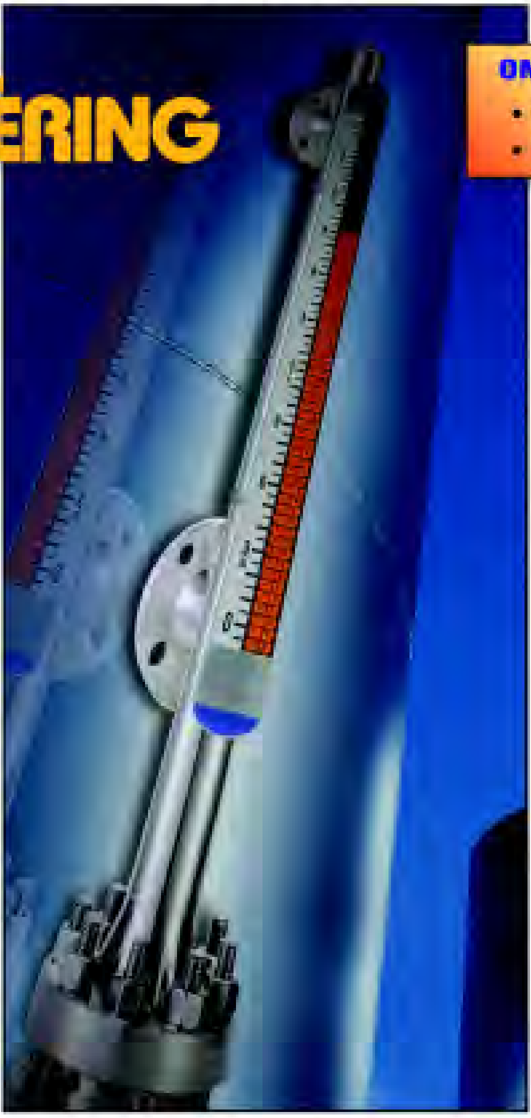

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COMING IN MARCH

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Cover: David Whitcher; Equipment photo courtesy of Orion Instruments

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Editor's Page

Prevalent plagiarism

One of the reasons that *Chemical Engineering* will have the honor of celebrating its 110th anniversary this year is its longstanding reputation for original, noteworthy and unbiased articles that cannot be found anywhere else. Our internal policies help protect the magazine's reputation from a number of threats, but not without relentless tenacity on the part of the editors. Much of that is to be expected, but what continually surprises me is the frequency at which we encounter blatant plagiarism.

Just last month, I received a manuscript that I ultimately discovered had been copied — entirely, it appears — from the Yarway Steam Handbook, which was ironically cited in the references section. Appalled by this discovery, I began reaching out to colleagues, readers, former authors and chemical engineering professors to see what they had to say about plagiarism. Many of their experiences were in line with those of my own, so my first reaction was to chalk the trend up to an overall decline in worldwide integrity. (Our audience is global, and this particular manuscript came from an author in Pakistan.) While integrity is certainly an issue, Sundar Madihally, associate professor of chemical engineering at Oklahoma State University, reminds us that now more than ever we must define what is — and is not — acceptable.

"I think the bigger problem is defining plagiarism in the international context," he says. "With the easy availability of resources on the internet throughout the world and significant influence of monetary benefits, it is always tempting as a human being to take the easy path. Also, in some parts of the world, authors may [consider that] plagiarism is not giving proper credit to the source. If they have [cited] the article where they got the sentences from, then they may perceive the incidence to not be plagiarism."

Whether the context is international or local, first-time writers or seasoned wordsmiths, admittedly, there are some instances where someone else said it best and it is worth repeating. With that in mind, here are some rules of thumb to follow when submitting a manuscript for publication:

- Above all, be clear about what is and is not original
- Unless there is particular impact in the exact words that the original source used, find your new words to summarize important information. Tom Overton, gas technology editor at *Power* magazine, aptly points out that this even applies to your own, previous work
- Use citations for bits of information (whether data or text) that you are summarizing and rewording yourself — unless it is already well-known
- If you must reuse someone else's work, employ quotes for short bits of text. A contrast in formatting, such as italics or indentation, is often preferred for lengthy or multiple paragraphs
- Before submitting a manuscript, obtain proper copyright clearance for anything more than a short quote, including tables, illustrations, diagrams and photographs. Give proper credit to each source
- Submit your manuscript exclusively and to only one publication at a time. Unfortunately, as noted by Robert Peltier, editor in chief of *Power* magazine, the practice of "shotgunning articles to multiple publications in hope of hitting one target is becoming more common these days"

At the end of the day, if personal ethics are not motivation enough, Ronald J. Willey, professor of chemical engineering at Northeastern University (Boston) leaves us with this. "My graduate school technical-writing teacher warned us early, 'If you attempt plagiarism, someday, sometime, it will catch up to you.' Surprising, 30 years hence, I have observed several times where he was right."* ■

Rebekkah Marshall

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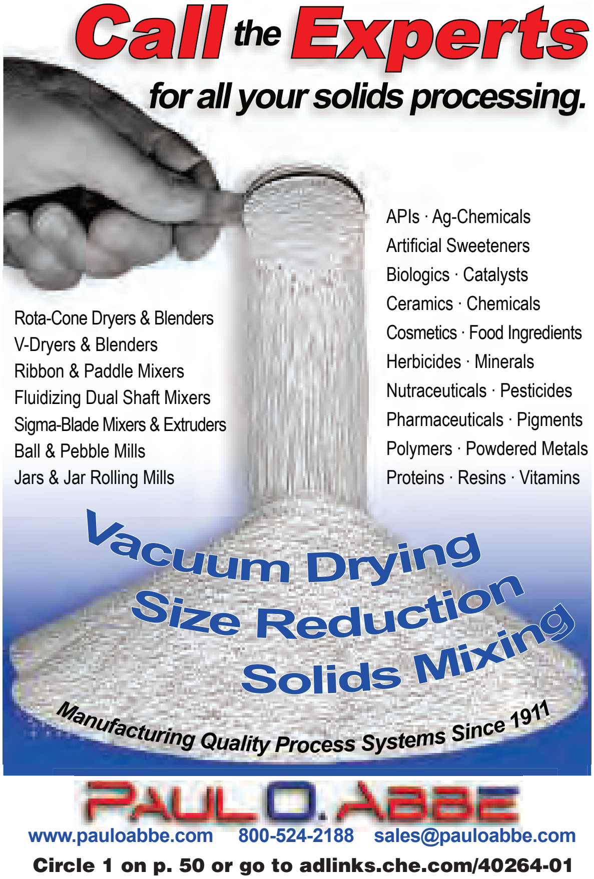
* We welcome your own comments on the online version of this article, which can be found under "Web Extras" at www.che.com.



Letters

Solutions for high-purity piping code

You ran a most illuminating article based on the new ASME code (July 2011, New Piping Code for High-Purity Processes, pp. 49–53), and I have been examining the welding industry's response to the new challenges. Recent years have witnessed an exponentially increasing demand for improvements in piping fabrication quality. The introduction of a new ASME piping code [*1*] is a formal recognition of this emerging requirement that has been stimulated mainly by the bioprocessing sector, but also by industries such as pharmaceuticals, semiconductors and food production.

An essential element in pipework is the production of welded joints, and the stringent inspection procedures imposed by ASME 33.1 apply as much to welded joints as they do to all the other fabrication processes involved.

A previous paper [*2*] highlighted more general aspects of the significance of the new code as it applies to the manufacture of high quality pipework. Producers of welding accessories designed specifically to meet the requirements have been quick to meet the new challenges imposed by ASME 33.1.

The problem. One of the fundamental requirements imposed during the welding of pipes is to prevent oxidation of the weld during the first pass. The welding torch provides inert gas coverage of the top of the fusion zone, but unless precautions are made there is no coverage of the weld root. The problem has always been recognized, and over the years a variety of solutions have evolved, some eccentric, others practical but largely ineffective.

While minimum standards are set for all fusion welding, the application to the pharmaceutical, semiconductor and food production sectors demands particularly high standards of cleanliness. Hygienic purity is the driving force for joints destined for use in pharmaceutical and food production. Elimination of particulate contamination is the crucial requirement in semiconductor manufacture.

With some semiconductor manufacturers producing chips with dimensions at the 32-nm level, and research going on at the 15-nm level, it is easy to see why the design, fabrication and maintenance practices required to ensure exacting purity requirements of their process fluid-distribution systems are of paramount importance.

In the food processing industries, statutory legislation and a plethora of litigation suits have forced plant manufacturers to introduce quality control levels previously considered unnecessary. Contamination introduced during fabrication is now unacceptable.

The solutions. The welding supply industry is leading the way in responding to the new demands imposed by ASME 33.1. See the online version of this article (at www.che.com under Web Extras) for references and specific solutions.

Michael J. Fletcher
Delta Consultants, U.K.

Postscripts, corrections

In the call for nominations for our Personal Achievement Award, we mistakenly said part of the activity must have taken place in 2007–2009. It should have said 2009–2011. The online version has been corrected.

Calendar

NORTH AMERICA

Biophysical Soc. 56th Annual Meeting. The Biophysical Soc. (Rockville, Md.). Phone: 240-290-5606; Web: biophysics.org/2012meeting
San Diego, Calif. **February 25–29**

3rd Annual ARPA-E Energy Innovation Summit. ARPA-E (Washington, D.C.). Phone: 512-697-8849; Web: arpa-e.energy.gov
Washington, D.C. **February 27–29**

NPRA Annual Meeting. National Petrochemical & Refiners Assn. (NPRA; Washington, D.C.). Phone: 202-457-0480; Web: npra.org/meetings
San Diego, Calif. **March 11–13**

Pittcon Conference & Expo. The Pittsburgh Conference (Pittsburgh, Pa.). Phone: 412-825-3220; Web: pittcon.org
Orlando, Fla. **March 11–15**

DCAT Week and 86th Annual Dinner. Drug, Chemical and Associated Technologies (DCAT; Robbinsville, N.J.). Phone: 609-448-1000; Web: dcat.org
New York, N.Y. **March 12–15**

Organic Process Research & Development. Scientific Update (East Sussex, U.K.). Phone: +44-1435-873062; Web: scientificupdate.co.uk
San Francisco, Calif. **March 19–21**

243rd ACS National Meeting & Expo. American Chemical Soc. (ACS; Washington, D.C.). Phone: 202-872-4600; Web: acs.org
San Diego, Calif. **March 25–29**

20th International Analytical Ultracentrifugation Conference. University of Texas Health Center (San Antonio, Tex.). Phone: 210-767-3332; Web: auc2012.uthscsa.edu
San Antonio, Tex. **March 25–30**

Management & Leadership Skills for Environmental Health & Safety Professionals. Harvard School of Public Health (Cambridge, Mass.). Phone: 617-384-8692; Web: https://ccpe.sph.harvard.edu/cms
Boston, Mass. **March 26–28**

Flexpo–Houston 2012. Chemical Market Res. (Webster, Tex.). Phone: 281-557-33209; Web: cmrhoutex.com
Houston, Tex. **March 29–30**

NPE 2012: The International Plastics Showcase. SPI: The Plastics Industry Trade Assn. (Washington, D.C.). Phone: 917-478-0754; Web: npe.org
Orlando, Fla. **April 1–5**

AIChE Spring Meeting & 8th Global Congress on Process Safety. AIChE (New York, N.Y.). Phone: 717-214-2200; Web: aiche.org
Houston **April 1–5**

Materials Research Society Spring Meeting & Exhibition. Materials Research Soc. (Warrendale, Pa.). Phone: 724-779-3003; Web: mrs.org/spring2012
San Francisco, Calif. **April 9–13**

24th Biennial ORCS Conference. Organic Reactions Catalysis Soc. (ORCS;). Web: orcs.org
Annapolis, Md. **April 15–19**

Milken Institute 2012 Global Conference. Milken Inst. (Santa Monica, Calif.). Phone: 310-570-4600; Web: globalconference.org
Los Angeles, Calif. **April 29–May 2**

A&WMA 2012 Annual Conference & Exhibition. Air & Waste Management Assn. (Pittsburgh, Pa.). Phone: 412-232-3444; Web: awma.org
San Antonio, Tex. **June 19–22**

EUROPE

In-Cosmetics. Reed Exhibition Group (Richmond, U.K.). Phone: +44-20-8910-7740; Web: in-cosmetics.com
Barcelona, Spain **April 17–19**

Analytica, 23rd International Trade Fair for Lab Technology, Analysis, Biotechnology & Analytical Conference. Messe München GmbH (Munich, Germany). Phone: +49-89-949-21474; Web: analytica.de
Munich, Germany **April 17–20**

Hannover Messe. Hannover Messe (Hannover, Germany). Phone: +49-511-89-31146; Web: hannovermesse.de
Hannover, Germany. **April 23–27**

Achema 2012. Dechema e.V. (Frankfurt am Main, Germany). Phone: +069-7564-277-296; Web: achema.de
Frankfurt am Main, Germany **June 18–22**

ASIA & ELSEWHERE

6th Jordan International Chemical Engineering Conference. Jordan Engineers Assn. (Amman, Jordan) and AIChE (New York, N.Y.). Phone: +962-6-5000900, Ext. 405; Web: jeaconf.org/JIChEC06
Amman, Jordan **March 12–14**

4th International Singapore Lipid Symposium. National University of Singapore, Center for Life Sciences (Singapore). Web: lipidprofiles.com
Singapore **March 13–16**

World Coal-to-Liquids (CTL) 2012 Conference. World CTL 2012 (Paris). Phone: +33-607-28-5247; Web: world-ctl.com
Beijing, China **April 17–20** ■

Suzanne Shelley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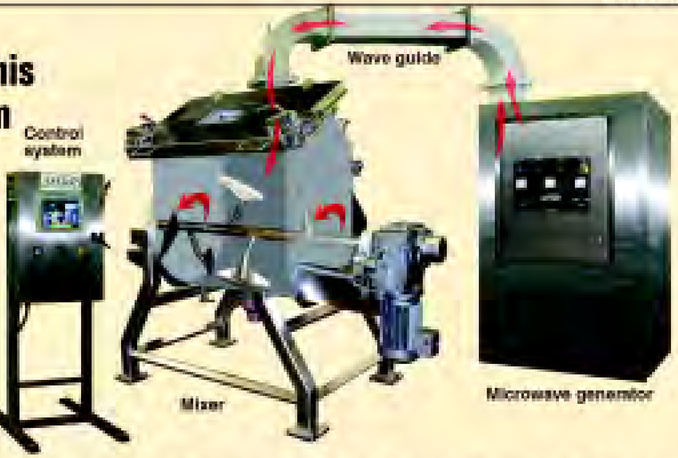
CHEMENTATOR

Edited by Gerald Ondrey

February 2012

Slash energy consumption with this microwave-heated mixing system



A microwave vessel heating system — said to be the first commercial, industrial-scale system of its kind — has been developed by Marion Mixers (Marion; www.marionmixers.com) in partnership with AMTek Microwave (Cedar Rapids, both Iowa; www.4amtek.com) with patent pending technology developed at the University of Northern Iowa (www.uni.edu). The combination of a mixer and microwave heater in a single piece of equipment has solved one of the biggest challenges in the microwave industry: to achieve uniform heat distribution, says Scott Jones, marketing manager at Marion Mixers. "This is achieved by Marion Mixers' unique paddle-style agitator and AMTek's patented microwave system."

The custom-made system (photo) is composed of three components: the microwave transmitter, waveguide and the mixing vessel. Electromagnetic energy is generated in the transmitter (mainly a magnetron) and travels through the waveguide and enters the vessel in a secure fashion protected by chokes. The material is heated by the microwaves inside the vessel chamber — tank or trough — and is discharged batchwise through a valve. Materials can be processed continuously.

In conventional drying, heat is transferred to the surface by conduction, convection or radiation, and into the interior of the material by thermal conduction. Moisture is initially flashed off the surface and the internal moisture then diffuses to the surface, explains Jones. This diffusion is often a slow process and requires high external temperatures to generate the required temperature difference. In contrast, microwaves excite the outer layer of water molecules on the material, and the inner part of the material is then warmed as heat travels from the outer layer inward. Most of the moisture is vaporized before leaving the material. This process is much faster, leading to shorter batch times with the potential to slash energy consumption by up to 50%, says Jones. Microwave heating also has the advantages of minimizing over processing, with no scorching, overheating or case hardening. Precise energy controls can be turned on and off instantly, which eliminates the need for warm up and cool down.

The commercial systems can be custom made in both horizontal (shown in photo) and vertical tank designs, with capacities from laboratory scale to hundreds of cubic feet or more in size. The systems are especially suited for moisture reduction and dewatering applications in the food, chemical, mineral, plastic and waste-to-energy industries.

Demonstration for integrated production of bioethanol

Oji Paper Co. (OJI; www.ojipaper.co.jp), in collaboration with Nippon Steel Engineering Co. (www.nsc-eng.co.jp) and the National Institute of Advanced Industrial Science and Technology (AIST; all Tokyo, Japan; www.aist.go.jp), has started pilot testing of a new, integrated process for making cellulose-based ethanol from fast-growing trees, such as eucalyptus. The pilot facility, located at OJI's Kure Factory in Hiroshima Prefecture, can process 1 ton/d of biomass and produce 250–300 L/d of ethanol. Testing of individual process steps will run through March, and preparations for fully integrated operation will begin in April.

The new process features: a new "mechano-chemical" pulping pretreatment step, which combines mild chemical treatment with grinding using a high-concentration refiner to overcome the low efficiency of enzyme saccharification and the high cost for electric power (the main problems associated with existing pretreatment technologies); a continuous saccharification and fermentation process, which also recovers enzymes for reuse; and a purification stage, which combines vacuum distillation, rectification and membrane dehydration. OJI is also developing a new fermentation enzyme as well as saccharification enzymes that are optimized for the raw materials.

This demonstration project is being supported (since 2009) by the New Energy and Industrial Technology Development Organization (NEDO) to establish efficient and low-cost technology to produce bioethanol from wood-based biomass, aiming to commercialize the ethanol production by 2020 under the five-year national program, Revo-

(Continues on p. 11)

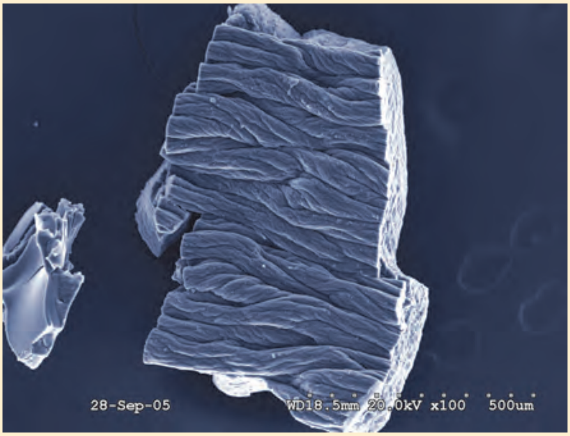
Fine polyester fibers

Teijin Fibers Ltd. (Tokyo, Japan; teijinfiber.com), the core company of the Teijin Group's polyester-fiber business, is commercializing a new version of its Nanofront, high-strength polyester nanofiber with a diameter of 400 nm — nearly half that of the first nanofiber the company launched in 2008. Nano-sized bumps on the surface of Nanofront increase frictional force and produce a surface area dozens of times greater than regular fiber, giving the material "outstanding" slip resistance, says the company. The fiber can be woven into cloths for industrial (abrasive cloth and filters) and clothing ap-

(Continues on p. 10)

Note: For more information, circle the 3-digit number on p. 60, or use the website designation.

Nano-engineered glass absorbs organic contaminants . . .



An organosilica material with unique structural properties due to its nanoscale architecture has been used to extract hydrocarbons and other organic contaminants from water. Marketed as Osorb by ABS Materials Inc. (Wooster, Ohio; www.absmaterials.com), the nanostructured glass represents a platform technology that has been customized by the company to address a host of water-treatment and remediation challenges, including removing hydrocarbons from produced water in oil and natural gas wells and removing chlorinated hydrocarbons from contaminated soil, among others.

Osorb contains the same stable Si-O-Si bonds found in glass, but its nanostructure gives rise to unique properties. The patent-protected Osorb formulations exploit interactions between functional groups at the molecular level to direct the organization of organosilica matrix at the macro level, explains Paul Edmiston, the inventor of Osorb, and chief scientific officer at ABS.

"During the final stages of production, Osorb is tensioned," says Edmiston, which he likens to compressing a nanomolecular spring (see micrograph). When the extremely hydrophobic Osorb material encounters organic compounds, the tension is relaxed, and the material swells. The organic materials are trapped in a matrix of nanoscale pores, from which they can be reversibly removed by vacuum or heat to regenerate Osorb material, which can then be reused. "Osorb is like a sponge for organic contaminants that has nanoscale pores to pull contaminants from water," Edmiston says.

The company has developed several ways to customize the Osorb material with functional metals or other molecular groups to address application-specific challenges. Among the first uses for Osorb to be commercialized is the in-situ removal of trichloroethylene (TCE) from soil. For that application, a slurry containing Osorb is injected into the soil of contaminated areas through a drill hole and traps molecules of TCE as they leach into the surrounding groundwater. In this case, the Osorb remains in the soil and continues to absorb TCE as it encounters it for years after injection.

. . . and serves as a nanoscale reactor for specific chemistry

Meanwhile, ABS Materials (see above) has used a library of basic Osorb formulations as platforms for a host of modifications to the material that allow it to function not only as a molecular trap, but also as a nanoscale reactor. Advanced forms of Osorb are being used to carry out catalysis, colorimetric detection and to perform commercially important synthetic chemical reactions.

A commercial example is the VOCEater system, which uses Osorb modified with palladium metal making it suitable for treating volatile organic compounds (VOCs) in water. The VOCEater system not only traps organic molecules but also reduces contaminants to salts through a catalytic hydrodechlorination reaction within the pore matrix. The VOCEater system can destroy pharmaceuticals, chlorinated solvents, pesticides, herbicides, endocrine disruptors and other compounds. Osorb has been shown to lower concentrations of hydrocarbons in produced water from over 200 to around 0.1 mg/L.

Osorb has also been used to treat flowback water from hydraulically fractured shale-gas wells (see Newsfront on pp. 14–16).

ABS Materials CEO Stephen Spoonamore envisions using the porous nanostructured glass materials with specific chemical groups on the inside of the pores to carry out important industrial reactions at ambient temperatures and pressures. Some examples under development are Suzuki coupling reactions (Pd-catalyzed joining of boronic acids with organic substituents and organic halides) and cycloaddition reactions (such as Diels-Alder reactions).

(*Continued from p. 9*)

plications. It is soft, stretchable, fits body contours, promotes cooling and has "excellent" absorbency and opaqueness, says the company.

A boost for biomass

Last month saw the start of a €5.1-million, 42-month research project dubbed Bioboost — Biomass-based energy intermediates boosting biofuel production. The project, partially funded under the E.U.'s 7th Framework Program, is coordinated by the Karlsruhe Institute of Technology (KIT; Germany; www.kit.edu) with participation from six research institutes and seven industrial partners, including Neste Oil, Grace, DSM and Chimar Hellas.

The project will focus on the decentralized conversion of biomass by fast pyrolysis, catalytic pyrolysis and hydrothermal carbonization to intermediate energy carriers, such as oil, coal or slurry. Based on straw, the energy density increases from 2 GJ/m^3 to 20–31 GJ/m^3, enabling a centralized gigawatt-scale gasification plant to process the carriers into biofuels, such as diesel, gasoline or kerosene, as well as chemicals (methanol, ethylene, propylene and plastics). Conversion plants in demonstration scale will enable the proof-of-concept and further scaling up to commercial size.

Wastewater treatment

Metso Corp. (Helsinki, Finland; www.metso.com) has commercialized an online measurement technology that enables operators of municipal and industrial wastewater-treatment plants to improve performance and reduce the amount of chemicals used in sludge-drying processes by up to one third. Said to be the first of its kind, the Metso Low Solids (LS) Measurement system is based on light-emitting diode and laser technologies. The device dearates, filters and measures the process solids, and also features self-cleaning capabilities to keep the optics clean for continuous measurement.

Already, feedback from users demonstrates the saving achieved by using the device. For example, a large

(*Continues on p. 12*)

The potential for reducing CO_2 in steel production

A reduction of CO_2 emissions of 50% can be achieved in steelmaking, using renewable carbon from biomass, according to The Australian Steel Industry CO_2 Breakthrough Program, an R&D program conducted by a team from: BlueScope Steel Research (Port Kembla, New South Wales; www.bluescopesteel.com), OneSteel Ltd. (Newcastle, New South Wales; www.onesteel.com) and CSIRO Minerals Down Under Flagship (Clayton South, Victoria; www.csiro.au). The team worked on both the integrated [blast furnace (BF) and basic oxygen furnace (BOF)] and electric-arc-furnace (EAF) steelmaking processes. It found that, of all of the applications considered for biomass-derived renewable carbon materials, pulverized fuel injection into the blast furnace has the highest potential for net reductions in CO_2 emissions. It concluded that the potential for using biomass is greatest for the integrated route, where direct or indirect emissions of CO_2 can be reduced by 50% or more.

Replacement of pulverized coal as the BF tuyere injectant is the application with the greatest potential for CO_2 mitigation. Pilot-scale testing by CSIRO has shown that charcoal can substitute for at least half of the solid fuel (coke or anthracite) used in sinter-making. Pulverized coal, or other injectants, are introduced into the raceway by means of a lance from a position within each tuyere. The blast velocity is about 250 m/s through the tuyere port, so that there is about 20 ms available to complete the combustion of the injectant in the raceway. Typical injection rates range from 100 to 200 kg/ton of hot metal (HM), or 20 to 40% of the total fuel usage. The blast temperature is typically 1,150 to 1,250°C.

On the other hand, because the EAF route uses scrap steel rather than iron ore, the CO_2 emissions per ton of crude steel are relatively low. The CO_2 associated with generating the electrical energy for the EAF process constitutes about 78% of the emissions in Australia. Thus, the opportunity for Australian EAF steelmakers to reduce CO_2 emissions using biomass is only about 6–12%, unless electrical usage can be reduced as a consequence.

Bioethanol

(Continued from p. 9)

lutionary Plan for Bio-base Fuel Technology, which was formulated in 2008 by the Japan's Ministries of Economy, Trade and Industry (METI) and Agriculture and Fishery (MAF). This program is also working on the selection, collection and transportation of biomass materials; the development of filamentous fungus for producing cellulose; and thermophilic enzymes for the simultaneous production of C_5 and C_6 sugars. When the program is completed in 2013, the next stage will be a commercial demonstration of the integrated process, with a 10-million-L/yr ethanol plant planned for southeast Asia or Australia.

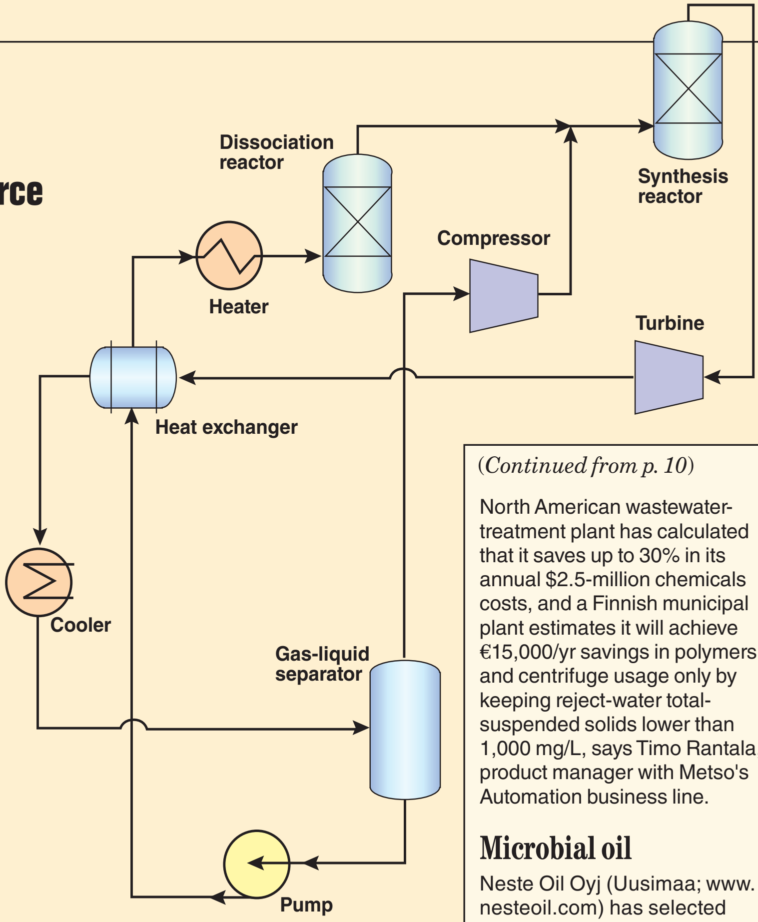
A simulation demonstrates the potential of NH_3 as a power source

Harnessing renewable energy by catalytic dissociation and synthesis of ammonia has shown promise for distributed power applications, according to a team from the Center for Energy at the University of Western Australia (www.uwa.edu.au), led by professor Dongke Zhang.

In the team's concept (flowsheet), solar or geothermal energy provides the heat required to dissociate ammonia into nitrogen and hydrogen gases via an endothermic reaction with a nickel catalyst. The gases are then synthesized back into ammonia using a ruthenium catalyst in an exothermic reaction. The hot synthesized gas is then used as the working fluid through a gas turbine using the Rankine cycle.

The ammonia produced as a working fluid is at 923K, whereas the ammonia needs to be heated to only 723K at the process pressure of 20 MPa for dissociation by the renewable source.

The team chose ammonia due to its low boiling point and a relatively low dissociation temperature. Heating ammonia to 723K is best done by means of a solar parabolic trough or a solar parabolic dish. The system has an efficiency of 25.4% producing 285 kW of electrical power with a molar flowrate of 100 kmol/h.



The team studied a simulation using two single reactors, one for the dissociation reaction and the other for the synthesis reaction. The temperature is maintained using an external fluid that is heated by the synthesis reaction to heat the dissociation reactor.

The team plans to develop a prototype to confirm the predictions regarding the efficiency and electrical power output.

Next-generation cross-coupling reactions

Professor Kenichiro Itami and coworkers at at Nagoya University (Nagoya, Japan; synth.chem.nagoya-u.ac.jp) have developed a new, next-generation type of cross-coupling reaction for synthesizing biphenyl compounds — one of the most important backbone structures for pharmaceutical and electronics chemicals. The chemists have developed the first nickel-catalyzed arylation reaction of the C–H bond of azole compounds using phenol derivatives. Prior to this development, such cross-coupling reactions required aryl halides or organometallic reagent (Ar–M) as a coupling partner, which added several reaction steps just to make the Ar–M; and the catalyst was based on costly palladium. The new approach uses readily available phenol derivatives as a coupling partner, and a nickel-based catalyst that is easy to make and less expensive than Pd-based catalysts.

The new catalytic system — $Ni(cod)_2$/dcype, where cod = 1,5-cyclooctadiene and dcype = 1,2-bis(dicyclohexylphosphino)ethane — is prepared from Ni and commercially available dcype, and is active for the coupling of various phenol derivatives, such as esters, carbamates, carbonates, sulfamates, triflates, tosylates and mesylates. By using the C–H/C–O biaryl coupling, a series of privileged 2-arylazoles — including biologically active alkaloids — were synthesized. Moreover, the researchers demonstrated the utility of this reaction for functionalizing estrone and quinine.

(Continued from p. 10)

North American wastewater-treatment plant has calculated that it saves up to 30% in its annual $2.5-million chemicals costs, and a Finnish municipal plant estimates it will achieve €15,000/yr savings in polymers and centrifuge usage only by keeping reject-water total-suspended solids lower than 1,000 mg/L, says Timo Rantala, product manager with Metso's Automation business line.

Microbial oil

Neste Oil Oyj (Uusimaa; www.nesteoil.com) has selected Neste Jacobs Oy (Porvoo, both Finland; www.nestejacobs.com) as its main partner for a pilot plant designed to produce waste-based microbial oil at its Porvoo refinery. It will be the first pilot plant in Europe designed to produce microbial oil for use in manufacturing renewable fuel from waste-based raw materials.

The plant will be used to develop microbial-oil-production processes and test various raw materials for producing microbial oil, such as straw and other agricultural residues, as well as industrial waste and residues. The plant will support Neste Oil's goal of using microbial oil to produce NExBTL renewable diesel in the future. The €8-million facility is due to be completed in the second half of 2012.

The production of NExBTL renewable diesel from microbial oil has already succeeded at the laboratory scale. The new pilot plant is an important step on the way to scaling up the technology to commercial capability, which is likely to be possible by 2015 at the earliest. ❑

Heavy-metal removal process joins advantages of two existing technologies

A newly demonstrated system for removing trace levels of heavy metals from water combines the positive traits of two existing technologies — chemical precipitation and electrowinning — while avoiding drawbacks of each. An engineering team at Brown University (Providence, R.I.; www.brown.edu), led by Joseph Calo, described the cyclic-electrowinning-precipitation (CEP) process in a recent issue of the *Chem. Eng. Journal*. CEP can efficiently reduce heavy metals concentrations, as demonstrated with mixtures of cadmium, copper and nickel, to below the levels set in U.S. federal drinking-water standards.

Chemical precipitation of metal ions has been used effectively to remove heavy metal ions from aqueous solutions, but the resulting toxic precipitate sludge presents a number of environmental and safety concerns for disposal. Electrowinning, in which metal ions are reduced and electrodeposited onto a cathodic surface, is another well-used technique for eliminating metal ions. A major drawback of electrowinning is that trace metals are typically present at concentrations too low to maintain good current-efficiencies.

The CEP process employs multiple precipitation-and-redissolution cycles that increase the concentration of metals to allow efficient electrowinning. The process retains toxic precipitates within the system, avoiding the disposal problem. In CEP, metal-laden water is drawn in, and precipitating agents (for instance, bases or sulfides) are added to react with heavy metal ions. The resulting "clean" water is siphoned off, and new metal-containing water is introduced. The precipitate is then redissolved by acidification, increasing the concentration of metal cations. The cycle is repeated until the concentration of metal ions is high enough to make electrowinning efficient.

The electrowinning stage uses a spouted particulate electrode (SPE) to convert cations into stable bulk-metal solids. The SPE allows operation at higher current densities and efficiencies than conventional flat-plate electrode cells, Calo says. The metals deposit on cathodic particles that can be further processed to recover individual metals. The Brown research team tested the CEP system with water containing Cd, Ni and Cu cations, and the concentrations were typically reduced to sub-ppm levels, Calo says.

The CEP process is scalable, Calo adds, and amenable to heavy-metal remediation and recovery applications. The group is working on adapting the approach to other heavy metals, such as mercury, lead and tin, and testing the system with aqueous solutions derived from contaminated environmental samples. ■

FRAC WATER REUSE

Reuse of water from fractured shale formations is growing, but success requires tailored treatment approaches to water chemistry


Kroff Well Services

FIGURE 1. Analysis of the microbial profile in fracturing flowback water is among the important considerations of treating water for reuse

Production of natural gas from shale formations presents key water management challenges. As the number of gas drilling operations continues to grow in shale plays around the world, and the practice of hydraulic fracturing (frac or fracing) of gas wells remains widespread, efficient water use strategies are becoming critical aspects of shale gas operations. Fresh water supplies for drilling and fracturing can be limited by regional climate, transportation logistics and competing industrial or agricultural uses. Meanwhile, wastewater disposal options are increasingly difficult because of high trucking costs, increased regulatory oversight and limited suitable underground sites.

Given the supply and disposal challenges, many gas producers are collecting water used in fracturing operations and treating it for reuse in a subsequent gas well. These water recycling approaches can reduce expenses, increase efficiency and help protect the surrounding environment; but several factors, including the complexity of shale gas production, the variability of water chemistries in different areas, and a host of regionally specific logistical issues, make one-size-fits-all approaches impossible. To arrive at viable individualized solutions, engineers need accurate assessments of local water chemistry and geology and a creative outlook toward applying available water treatment technologies.

Treatment and reuse of water is becoming the dominant approach now, acknowledges Dave Grottenthaler, general manager of Kroff Well Services Inc. (Pittsburgh, Pa.; www.kroff.com). "Most gas producers are reusing a large portion of their flowback water — as much as 90% — for fracturing other wells," he says.

Experts agree that the wide range of proven, available water-treatment technologies allows water from fractured shale wells to be recycled effectively. "The prospects for treating flowback water to a recyclable condition from fracturing operations in shale-gas formations appears very good," remarks Dave Manz, water engineer at Oasis Filter International Ltd. (Calgary, Alta.; www.oasisfilter.com). "Today's treatment-and-reuse strategies are economically competitive with currently used deep-well disposal methods."

"Whether you are going to reuse the water [from fractured wells] or discharge it to municipal facilities, some degree of water treatment is required," says Bob Kimball, process engineer at CDM Smith (Cambridge, Mass.; www.cdmsmith.com). "Although research and development continues to improve water treatment, we focus on applying current technologies in creative ways," says Kimball.

Water-based fracturing fluid

The current expansion of operations to extract natural gas from shale formations is largely the result of improvements in hydraulic fracturing technology, and refinement of horizontal drilling practices. Together, the two factors have made it economically viable to locate gas operations in shale formations that were inaccessible or uneconomical in the past. Hydraulic fracturing — a technique that has been employed in the oil and gas industries since the 1950s — is a well-stimulation technique in which fluid is pumped at high pressure into the formation to create cracks in the target rock. The fractures are held open by a proppant (usually sand) that is included in the fracturing fluid to facilitate gas escape into the wellbore.

Most industry estimates say that typically around 2–4 million gal of water (with additives) are required to drill and fracture a single shale well, although the volume can vary widely (1–10 million gal), depending on specific circumstances. Water accounts for 99.5% of the volume of conventional fracturing fluid, to which a customized mixture of chemicals is added (Figure 2). The additive mix in fracturing water varies widely depending on the permeability of the shale formation and other factors, but typically includes biocides, scale inhibitors and friction-reducing compounds designed to allow the water to maintain a high velocity in the well. The high-velocity water better maintains the proppant material in suspension while transporting it to the fracture network, explains Manz.

To effectively select the correct combination and concentration of fracturing fluid additives and proppants, engineers need information about the formation's geology — porosity, perme-

FRACTURING WITH LPG

GasFrac Energy Services Inc. (Calgary, Alta.; www.gasfrac.com) is among a small minority of companies avoiding the use of water as a fracturing fluid altogether. GasFrac uses gelled liquefied petroleum gas (LPG) in place of conventional frac fluids. Although the use of LPG as a frac fluid avoids many of the supply and treatment issues found with water, GasFrac chief technology officer Robert Lestz says the impetus for using LPG was really to increase well productivity.

As a frac fluid, LPG has several advantages: whereas a large portion of water-based frac fluids remain in the reservoir, where they can contribute to reduced performance, virtually all of the LPG is recovered along with the produced natural gas (also oil). The capillary effect of the LPG inside the fractures in the rock, coupled with the LPG's solubility with natural gas and miscibility with oil from the formation, keep the shale pore openings clear and allow higher well productivity. Also, since mineral salts are not soluble in LPG, they are not conveyed back to the surface as they are with flowback water. With LPG, a lower overall volume is needed to frac each well, and handling LPG plays into core competencies of the oil and gas industry.

Fracturing with LPG presents safety risks. To mitigate them, Lestz says that nitrogen gas blankets are used to isolate the frac fluid from air, and all operations at the wellhead are completed remotely during fracing, so that no personnel are close to the wellhead. ❑

ability, pressure and temperature gradients, mineral content — and about the chemistry of the water initially used in the fracturing job. Ideally, fracturing water should be high-quality, disinfected water that is free of chemicals that could interfere with those used in the fracturing or that might damage the formation, Manz says.

"The best water for fracturing would essentially be clean salt water with low levels of divalent cations and low bacteria load," Grottenthaler suggests. Kroff has completed studies that indicate using fracturing water with higher levels of chlorides (~80,000 ppm) boosts gas production at the well compared to fresh water with total dissolved solids (TDS) at low (~400 ppm) levels.

In addition to additives, flowback water contains some natural formation water that has been in contact with the reservoir formation for millions of years and thus contains minerals native to the reservoir rock.

Although water is by far the most commonly used fracturing fluid, other techniques can avoid some of the water use-and-treatment challenges associated with water-based fracturing fluids. For example, using liquefied natural gas as a fracturing fluid presents unique advantages, but also safety challenges (see box, top of page).

Flowback water

After a fracturing treatment on a gas well, the pumping pressure is relieved and fracturing fluid, mixed with water from the rock formation, begins to flow back through the well casing to the wellhead. In treatment-and-reuse scenarios, this is the water that will serve as the basis for fracturing fluid for the next job. Otherwise, this water must be treated before discharging to a municipal water treatment facility, because such plants are not designed to handle typical fracturing-flowback water.

In most cases, wells recover between 20 and 40% of the water that was pumped into the well for fracturing, although the amount can be higher. The portion of the fracturing water that does not return occupies pores and fractures in the shale formation that have been vacated by the gas.

The well flowback rate is highest initially and then decreases. There can be wide variation in flowback rates across geographical locations and because of operator bias. A typical flow profile could be the following: the flowback rate over the first 1 to 5 days after fracturing is around 100 to 150 bbl/h, and recovers 10–25% of the initially injected water; then over the next 10 days, the rate decreases to 20 to 60 bbl/h and recovers another 8 to 12%. From 15 to 30 days past the end of the fracturing job, 5 to 10 bbl/h might flow, and for one to three months past, 10 bbl/d may be recovered.

Treatment challenges and goals

While most flowback water from shale fracturing operations shares some characteristics, the physical and chemical variations in water from different geologic basins and even within regions in a single basin are much more pronounced. For example, levels of total dissolved solids (TDS) can range from 5,000 ppm to greater than 200,000 ppm. Other variable constituents that need to be considered for treatment operations include the following: hydrocarbons (oil and grease), bacteria, suspended solids, soluble organics, iron, calcium, magnesium, trace constituents (benzene, boron, silicates) and possibly others, such as naturally occurring radioactive materials. As an example of the variability, Kimball shared data comparing levels of common constituents in water from four different fractured wells (Table 1). Moreover, flowback volume and water properties can vary throughout the lifetime of the well.

So the main challenge for engineers is structuring water treatment strategies around these variations on a regionally specific basis. Shelley Martin, a scientist in the office of research and development at the Dept. of Energy's National Energy Technology Laboratory (Pittsburgh, Pa.; www.netl.doe.gov), says regional differences in water properties demand a localized approach to treatment technology. "The goal is to tailor water treatment technologies to the particular region where the water is coming from," Martin says.

CDM Smith's Kimball explains that generally, water treatment goals for reuse are the cost-effective removal of hydrocarbons, friction reducers and other polymer additives, as well as the elimination of inorganic scale-forming compounds and bacteria.

Among the major fracturing water challenges in shale wells is inhibiting scale accumulation on the surface of the formation, which can impede gas flow and permeability, and reduce production. In the view of Kroff's Grottenthaler, "The number one problem for water treatment is barium sulfate, because its formation is virtually irreversible." Barium sulfate can also form particulate matter that interferes with the proppant. Following barium sulfate, the next biggest problem for water treatment is reducing bacteria, Grottenthaler says. Dealing with iron sedimentation and other scale-forming substances, such as calcium and magnesium are also important.

Among the viable technologies for removing organics are oil-water separators, dissolved air flotation, chemical oxidation, biological processes, activated carbon treatment, walnut shell filters, organo-clay adsorbents and air strippers. For inorganic treatment, oftten-used technologies are chemical precipitation, lime/soda softening, clarifiers, ion exchange membranes, sand filtration and others.

Describing an example treatment scheme for incoming fracturing flowback water, Kimball says that the oxidant ClO_2 could be used to break oil emulsions, destroy friction reducers and kill bacteria. Hydrocarbons could then be removed by dissolved air flotation, where fine bubbles diffusion floats oil, grease and total suspended solids (TSS). A liquid-phase, activated-carbon step can remove hydrocarbons and organics. A chemical precipitation system could then remove inorganic scale-forming compounds, and the sludge could be dewatered and disposed of. A sand filter could then remove remaining TSS before reuse.

Once suspended solids, organics and bacteria are removed, TDS can be reduced with a host of other technologies, depending on concentration. Among the potentially useful technologies are ion-exchange membranes, reverse osmosis filters, evaporators and crystallizers, Kimball says.

In addition to configuring technologies to address the local water chemistry, gas producers need to consider the format that will house the technologies. Water treatment equipment can be constructed in completely mobile setups, in semi-permanent (modular-type) operations or in fully permanent facilities (centralized treatment).

"The volumes of flowback water are large enough that semi-fixed treatment facilities are economical," says Grottenthaler. Semi-permanent water treatment facilities that would remain in place for several months to serve multiple wells in a particular area will be more common, he predicts.

Water analysis

Optimized water treatment designs are based on accurate, comprehensive analyses that identify water quality, scaling potential, microbiological profile, compatibility with geological formations, and other factors.

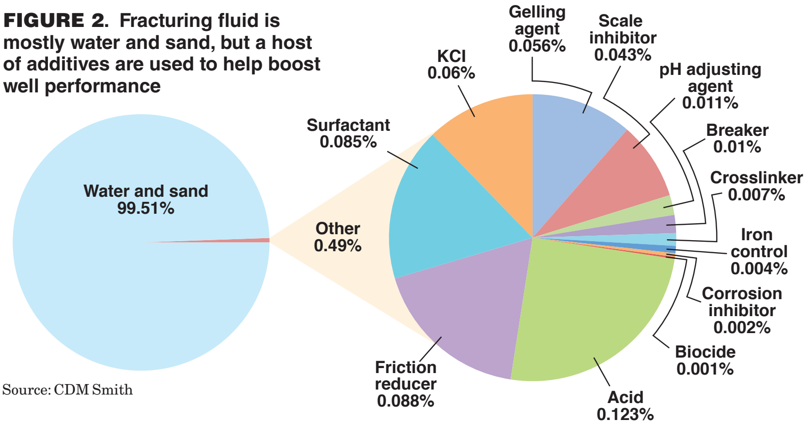
The comprehensive approach to water analysis taken by Kroff represents an example of how data can influence the design of treatment-and-reuse schemes. Kroff has assembled a database of pre-fracturing and fracturing-flowback waters from many shale formations in different regions. The company tracks changes in water chemistry over time by collecting flowback water samples every 500–1,000 bbl of post-fracturing flowback. Kroff uses the sequential flowback analyses to recommend strategies for compatible flowback water reuse, including plans for water remediation, blending fresh water and optimizing the design of fracturing fluids.

TABLE 1. EXAMPLES OF FRACTURING FLOWBACK WATER CHEMISTRY SHOW WIDE VARIATION

Parameter	Frac1 (mg/L)	Frac2 (mg/L)	Frac3 (mg/L)	Frac4 (mg/L)
Barium	7.75	2,300	3,310	4,300
Calcium	683	5,140	14,100	31,300
Iron	211	11.2	52.5	134.1
Magnesium	31.2	438	938	1,630
Manganese	16.2	1.9	5.17	7.0
Strontium	4.96	1,390	6,830	2,000
TDS	6,220	69,640	175,268	248,428
TSS	490	48	416	330

Courtesy of Bob Kimball, CDM Smith

FIGURE 2. Fracturing fluid is mostly water and sand, but a host of additives are used to help boost well performance



Source: CDM Smith

"Our proprietary fracturing-fluid calculator incorporates the pre-fracturing water analysis and historical flowback results from that region and provides recommendations for component dosages based on the geology," the company says.

Another focus of the analysis is microbiology. Kroff analyzes microbiological concentrations in water samples, and can supplement the testing with more extensive analysis of sulfate-reducing bacteria, acid-producing bacteria and total aerobic bacteria counts. Knowledge of the bacterial profile allows Kroff to select the correct microbiocides at the optimal application dosage for the particular fracturing fluid.

Simulating the geomechanical behavior can also influence the selection of friction-reducing compounds and their amounts. Kroff has a simulation loop to help optimize the friction-reducing performance of the water.

Future ideas

As shale gas drilling expands over the next several years, a number of shifts are likely to develop. First, an increase in the number of wells will result in a greater volume of both fracturing flowback water and produced water from the wells. As the total volume of water requiring treatment grows, the proportion of produced water will also increase significantly, says Grotthenthaler. Larger volumes of produced water, along with efforts to improve efficiency and lower costs, will stimulate producers to refine their techniques and engineers to continue improving treatment technologies. For example, Grottenthaler believes gas producers will make greater use of selective ion-exhange media, since it is likely that some ions in fracturing water actually help gas production, while others can be problems.

Looking forward, a host of other ideas may also come into greater practice. For example, a number of engineers are investigating the use of acidic water flowing from abandoned coal and metal mines (acid mind drainage; AMD) as a source of frac water. Use of AMD in frac water can help mitigate the problems caused by the drainage itself while also lessening the pressure on other fresh water resources.

Frac water modifications may also become more widespead, as companies seek ways to increase gas production. An example is dissolving N_2 or CO_2 in the frac water to boost production. ■

Scott Jenkins

MEMBRANES FOR GAS SEPARATION

Newly developed membrane materials bring lower costs and big benefits to gas separation applications


UOP


UOP

FIGURE 1. Separex membranes from UOP are for use in the CO_2 removal market

FIGURE 2. This FPSO employs UOP's Separex systems for CO_2 removal offshore. Benefits of membrane technology in these applications include lighter weight, smaller footprint and fewer moving parts for simplified operation and maintenance

Limited budgets and the need for high performing operations are causing chemical processors and petroleum and gas refiners to seek out the best bang for their capital dollar. And when it comes to gas processing operations, such as carbon-dioxide removal, hydrogen purification and air separation, experts say today's membrane technologies compare favorably to other available systems and will continue to do so for years to come as research and development work is currently underway for future applications.

Membrane technology has been available since the mid 1980s, but has become more prevalent in gas processing operations as new developments have helped it filter into more applications. "New materials allow the membrane modules to operate at more extreme parameters, such as higher temperatures and pressures, and there's been modifications to the chemistry of making the membrane polymer so that they are able to withstand increasingly harsh conditions," says David Marchese, director of sales and marketing for the Medal division of Air Liquide Advanced Technologies (Newport, Del.).

But perhaps the biggest attraction to membranes over other gas processing systems, such as amine plants for CO_2 removal and pressure-swing adsorption (PSA) technology for hydrogen purification, is that membranes do not have many moving parts.

CO_2 removal

When it comes to CO_2 removal, projects are usually onshore or fixed offshore production, storage and offloading units (PSOs) or offshore floating production, storage and offloading units (FPSOs), so the mechanical simplicity of membranes provides several economic advantages in these types of projects over other technologies, such as amine plants, which require a significant level of skill to operate and maintain.

"In onshore projects, the drivers behind membrane use include speed to delivery, being able to operate in remote environments with lower manpower and skill requirements and overall operating efficiencies that membranes bring to the table over other technologies," says Mark Schott, director of business development for the gas processing business of UOP LLC (Des Plaines, Ill.), a Honeywell company.

And in offshore CO_2 removal projects, there are similar benefits, according to Schott. "When talking about either fixed offshore PSOs or FPSOs, then the size and weight requirements of membrane systems are significantly lower than competing technologies for CO_2 removal and therefore allow the customer to invest less money on the platform or fit gas treatment equipment on top of FPSOs without having to make the ship bigger," he says. "So there are very significant economic benefits to the customer by being able to use a membrane that drives them toward lower capital spending, lower overall costs, lower weight and smaller footprints for those applications" (Figures 1 and 2).

For this reason, mechanically simple membranes made with new and improved materials that allow them to work in harsh environments and conditions have filled a certain niche in the CO_2 removal market where speed to market and operating in difficult locations and environments like deserts, cold climates and jungles are necessary.

While there are certainly benefits

Air Liquide

FIGURE 3. The air-separation modules shown here are for a nitrogen-production unit

Air Liquide

FIGURE 4. A complete membrane system for H_2 is shown here

to membranes in CO_2 removal applications, Schott warns that there is a downside in that membranes are separators and not purifiers. "For every bit of CO_2 you remove, you also remove a small amount of methane so you end up with a permeate stream, which may require multiple stages to increase methane recovery."

And depending upon the allowable specifications for percentage of CO_2 in the feed, membranes can't always reach the lower levels achieved by solvent technologies. So the necessary product specification for the gas must be taken into account when considering membrane technologies in these applications.

However, as membrane technology providers continue to improve their products, this may not be an issue for much longer. "We continue to improve the membrane productivity of our Separex line of membranes for CO_2 removal so that the capacity of gas separation you're able to get out of a single membrane element increases while at the same time we are working toward reducing the cost of putting that membrane inside a system," says Schott.

In addition, Air Liquide has developed a hollow-fiber membrane product that purifies biogas, landfill and digester gas, where it removes CO_2 from methane associated with off-gasses from these applications. "This allows us to upgrade biogas into pipeline quality or natural gas used for CNG or LNG," says Marchese. "This is a very novel application for membranes."

Hydrogen purification

In the oil and gas industry, the biggest challenge right now is being able to meet tougher fuel specifications, which requires the recovery of hydrogen from off-gas streams. Currently membrane systems and pressure swing adsorption (PSA) units are the existing options. PSAs will provide higher purity, but lower pressure products while membranes give higher pressures, but lower purity levels, so the technology of choice depends upon the source of the stream and whether pressure or purity is the main concern.

However, improvements to hydrogen purification membranes may also help boost the use of membranes in these applications. "With regard to hydrogen purification from hydrocarbons, Air Liquide introduced a polymer that allows for greater contaminant resistance, so that when we put this polymer in a hydrogen refinery or chemical processing facility, we expect longer operating life from that particular membrane module," says Marchese.

And with a growing demand for new hydrogen purification technologies in the refining sector, membrane technology proves to be a good, incremental way to extract hydrogen from existing hydrocarbon streams as opposed to building a new hydrogen plant or stream methane reformer, he continues. Air Liquide's hollow-fiber hydrogen purification membrane can be employed to recover the hydrogen in that off-gas so it is not flared or wasted (Figure 4).

In both hydrogen purification and CO_2 removal, membrane providers are working on improvements in materials that would provide even more benefits. UOP, for example, is focusing on new materials, new formats and hybrid or mixed matrix membranes. "This means putting molecular sieves or other inorganic materials inside a polymer membrane to improve the performance of that polymer membrane," says Schott. "You can make inorganic membranes, but they tend to be very brittle and expensive, so this hybrid approach of polymer plus inorganic materials could be a bridge to better performance at lower cost than a straight inorganic membrane."

Air Liquide, too, is making improvements in the way it manufactures membrane modules and is exploring different materials to create the "next generation membrane." These improvements, says Marchese, are geared toward improving the separation efficiency and selectivity to make processes even more efficient.

The future of membranes

In addition to improving upon current membrane technology for existing applications, some companies are working on research and development aimed at creating entirely new membranes for applications in the foreseeable future.

Two of the areas driving this developmental trend are large-scale alternative-energy projects and the need for hydrogen in making cleaner fuels from refining petroleum in an effort to meet the even-more-stringent clean-air standards expected in the future.

GAS SEPARATION MEMBRANE SERVICE PROVIDERS

Medal division of Air Liquide	www.medal.airliquide.com
Air Products and Chemicals	www.airproducts.com
Pall Corp.	www.pall.com
Praxair	www.praxair.com
UOP, a Honeywell Company	www.uop.com

For this reason, oxygen-transport membrane technologies are being examined closely by companies like Praxair (Danbury, Conn.), which is focusing on developing inorganic membranes for some of these applications. “These membranes will be made of ceramic materials and will be perfectly selective for oxygen,” says Dante Bonaquist, executive director for R&D at Praxair.

The first technology, which has recently become commercially available, is meant to replace small-scale packaged oxygen cylinders for industrial applications. Praxair developed oxygen transport membranes made of ceramic materials that can be used to produce relatively small volumes of high-purity, high-pressure oxygen as a replacement for packaged oxygen, in which oxygen is sourced from liquid oxygen and packaged in cylinders at high pressure and distributed (Figures 5 and 6). “The oxygen transport membrane can be used in a small scale unit that can be plugged into the wall and run on electricity to produce oxygen at high pressure and high purity at the customer’s point of use so they wouldn’t have to change cylinders, but instead would have oxygen continuously supplied from this unit,” says Bonaquist.

The biggest benefit here, other than convenience, is that the unit provides “an absolutely constant oxygen purity level” for relatively small applications. One of these units would replace up to ten full-size oxygen cylinders per month.

The second application for oxygen transport membranes is for the production of synthesis gas (syngas) and oxy-fuel combustions, which moves the technology into the large-scale and very large-scale arena. Traditionally for the production of syngas or oxyfuel, a separate oxygen supply system was needed, such as a vacuum pressure-swing adsorption unit for producing oxygen, which is then fed into the process to make the syngas or into the combustion process.

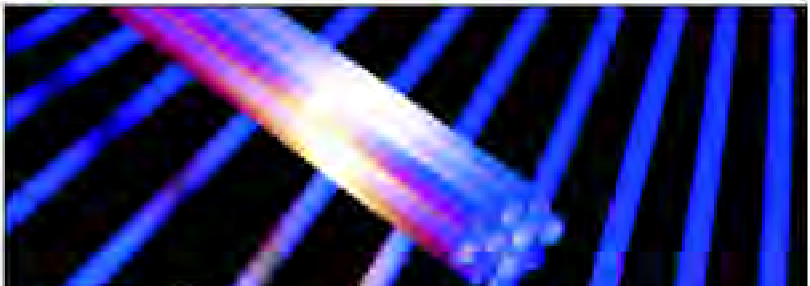
With Praxair’s development, the oxygen transport membranes could be integrated into the syngas production unit or the oxyfuel combustion application so that there is not a separate oxygen supply system. “Oxygen is taken out of the air, pulled across the membrane and reacted with the methane to create the driving force to deliver the oxygen,” says Bonaquist. “So we are effectively integrating the oxygen supply with the application itself to eliminate the need for separate oxygen supply. This technology is expected to be more readily available within the next year.

Similarly, Air Products and Chemicals (Allentown, Pa.) is working on developing and scaling up its ITM (Ion Transport Membrane) technology for the production of oxygen and syngas.

ITM technology uses a ceramic material which, under pressure and temperature, ionizes and separates oxygen molecules from air. No external source of electrical power is required in this process. ITM technology has the potential to produce oxygen more economically and efficiently, decrease the oxygen plant footprint, and also decrease the cooling water requirement for most large oxygen-demand applications, says Mark Kappes, global business manager of Air Products Prism membranes.

“Our next step in ITM Oxygen technology is to have an operating 100-ton/d intermediate-scale test unit on-stream by the end of 2012,” he says.

While improvements to existing hydrogen purification membranes are underway, developments in the area of entirely new materials for these membranes are also on the horizon, which is becoming more and more necessary due to requirements for cleaner fuels



Praxair

FIGURE 6. Praxair's tubular ceramic membranes can be reliably manufactured, provide excellent seal capability and are robust to the thermal, chemical and pressure stresses experienced in operation

Praxair

FIGURE 5. Praxair's StarGen Ultra-High-Purity Oxygen Generator (prototype pictured) fulfills the need for a consistent supply of oxygen without the need for cylinder change-outs and inventory maintenance, providing a virtually continuous supply on demand

and the need for alternative energy.

Praxair is currently working on an inorganic hydrogen transport membrane. The new membrane would be comprised of a metallic coating on a ceramic substrate and would be perfectly selective for hydrogen. It is intended to purify hydrogen from an impure source, such as a gas stream in a chemical, petrochemical or refining process where the byproduct stream contains some hydrogen and the goal is to recover hydrogen as a pure stream. "The hydrogen transport membrane we have in development would significantly lower the cost of purifying hydrogen from a variety of sources," says Bonaquist. "It would also improve upon economies enough to allow higher hydrogen purities to be achieved and be able to recover hydrogen from a broader variety of source gases than a PSA can handle. Functionally, it would do the same thing as a PSA but it would expand the capabilities and improve the quality."

Pall Corp. (Port Washington, N.Y.) is developing a membrane based upon palladium alloys to provide an economic high-flux, high-selectivity hydrogen membrane for use where traditional, polymeric hydrogen purification membranes could not withstand the high temperatures. Unlike the commercially available thicker palladium alloy tubes or foils, Pall is developing thin-film composite membranes using its own tubular porous supports to lower the cost of hydrogen purification.

The company is initially starting with small scale modules that can address current customer needs. These membrane modules will hold the palladium alloy composite membrane tubes and will be about 15 in. long. The modules will be designed to produce from 10 to 200 L/min of hydrogen, depending on the number of membrane tubes and operating conditions.

This solution would consist of the pressure vessel with the membranes inside and would be integrated into the customer's existing system. Targeted applications for this product would be as a hydrogen purifier for high temperature applications where it could be combined with a reformer to create a pure hydrogen-gas stream that could be fed to a fuel cell. "One of the targeted markets for this combination of hydrogen purifier, reformer and fuel cell is in back-up power applications, such as supplying power to a phone tower," says Sean Meenan, senior vice president, marketing, fuels and chemicals, with Pall. From there, the company will scale up the size of the high-temperature separation device for use in other applications.

In addition to working toward increasing the physical size of the membrane device, Pall is also working on making these palladium membranes tolerant of components found in gas streams in some chemical and petroleum refinery processes. "We would like to make them more tolerant of sulfur compounds so they may be used to separate hydrogen from CO_2 in high-temperature gas streams downstream of gasifiers and to recover hydrogen from high-temperature purge-gas streams."

No matter how they are approaching it, those developing these new membranes say both new oxygen and hydrogen membrane technologies are expected to play a huge role in a future where cleaner fuels are required and the global demand for energy will only continue to grow, necessitating the need for syngas and oxyfuel as ways to supplement energy derived from petroleum products. ■

Joy LePree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Fractionation Column

Graduate students' journey to the U.S.

Mike Resetarits is the technical director at Fractionation Research, Inc. (FRI; Stillwater, Okla.; www.fri.org), a distillation research consortium. Each month, Mike shares his first-hand experience with *CE* readers

I was whining to myself, and to anybody who would listen, about the 20 to 30 projects that I have on my desk at all times. Then I met Ms. Regina Henry, who is the coordinator of immigration at Oklahoma State University (OSU). Together, Regina and I counted her active projects. They totaled 460. Regina, two assistants and two part-time assistants are currently helping 460 foreign-born students to study and stay in the U.S. Her office is large, but not too tidy. How could it possibly be tidy?

The job that is done by Regina, her colleagues and her counterparts in other universities across the U.S. is of critical importance to U.S. engineering — especially chemical engineering. Some chemical engineering jobs require advanced degrees, M.S. and Ph.D. degrees. This is particularly true in R&D organizations, including FRI. Unfortunately, the vast majority of American-born B.S.Ch.E. graduates leave academia for industry. It is easy, these days, for such graduates to get offers like $65,000/yr to put their books away — an attractive prospect to people who have made $10,000/yr for four years, while waiting tables and cutting lawns to work their way through colleges.

Very fortunately, there are lines of foreign-born and foreign-educated B.S. graduates eager to fill the U.S. graduate school gaps, especially in chemical and other engineering programs. OSU's chemical engineering graduate-school program is strong. Enrollment the last three years is higher than it has been in 25 years. Roughly half of these students are pursuing M.S. degrees; the rest, Ph.D.s. Of all of these, 90% are foreign born and only 33% are female.

So, why do they leave their families and friends and travel thousands of miles to get here? Answer: Many of them want to stay here, and to stay here they need to study here. A degree and an advanced degree from an Asian or Middle Eastern university is just as valid as a degree from a U.S. university, but, it is very difficult to secure a job in the U.S. based solely on a foreign degree. About once a week I receive a resume from afar. The FRI budget will not allow me to fly anybody 10,000 miles for a 45-minute interview.

American students cannot imagine the hurdles that foreign students face as they pursue U.S. graduate educations and then jobs and then green cards. Just to get to the U.S., the following steps are required: Obtain an I-20 form from the university. Visit the U.S. embassy. Obtain an F1 student visa at a U.S. embassy/consulate (funding availability from the university increases the probability of success from about 25 to 50%).

Once a student arrives in the U.S., more paperwork needs to be filled out in the International Student Office; immunizations and orientations are required. While studying, curricular practical training (CPT) allows a student to perform intern work for up to 12 months. Upon graduation, a graduate has 90 days to find a job or an instructor who will sponsor a volunteer, to avail a one-year optional practical training (OPT) work permit that comes with the degree. From there, an H1-B visa, which is always employer-sponsored, needs to be sought and approved. An H1-B visa can extend the work for up to six years. Within six years, the "student" must apply for a green card — or return home. A green card, and permanent residency, is the ultimate prize for many students after all, but the backlog for green cards is long. The wait can be anywhere from three to 9 years.

There is, of course, a cost to employers who hire foreign-born graduate students. Regina does not like the word "cost," preferring the word "investment." I agree with her. To all of the foreign students who read this editorial: We need you and we thank you. ■

Mike Resetarits
resetarits@fri.org

CHEMICAL ENGINEERING
FACTS AT YOUR FINGERTIPS

Department Editor: Scott Jenkins

pH electrodes and temperature dependence

Acidity is a critical parameter in the chemical process industries, and accurate measurements of pH can be important indicators for process quality control. A refreshed understanding of pH measurement can help to overcome process-specific challenges to measurement and to select the pH measurement devices that best meet the needs of the process. Further, a review of the temperature dependence of pH measurement devices can help instrument selection.

pH review

pH is defined as the negative logarithm of the molar concentration of hydrogen ions in a solution. A water solution is acidic or basic depending on whether a net excess of H^+ (acidic) or OH^- (basic) is present. The reality of proton-containing cations in solution is more complicated (hydronium ions, Zundel cations and Eigen cations exhibit complex solvation structure), but the definition is useful for industrial determinations of acidity or alkalinity.

$$pH = -log\,[H^+] \qquad (1)$$

Generally, free H^+ and OH^- ions combine to form neutral water, but there are always small amounts of each of these ions in water. The two concentrations are related by the dissociation constant of water, K_w.

$$K_w = [H^+][OH^-] = 1.0 \times 10^{-14} \text{ (at 25°C)} \qquad (2)$$

All three terms can be expressed as logarithmic terms, and can be related to each other in the following way:

$$pH + pOH = pK_w \qquad (3)$$

Since pH is expressed on a logarithmic scale, each unit of pH corresponds to a factor of 10 in concentration. For example, pH 4 wastewater has 100 times the acid content of a water solution at a pH of 6, and neutralization will require 100 times the amount of base to combine with the acid.

pH sensors

In general, pH sensors consist of a silicate-glass measuring electrode that has been rendered pH-sensitive by the addition of alkali metals, as well as a reference electrode (commonly based on silver and silver chloride) to complete the circuit (Figure 1). The measuring electrode can be thought of as a battery that produces a millivolt output that varies with changes in the hydrogen ion concentration inside and outside of the glass bulb of the electrode. When the pH of the process solution is the same as that of the reference electrolyte, no electrical potential exists across the glass. In acidic solutions, negative potential exists; the opposite is true for basic liquids.

Devices that measure pH use the Nernst equation (Equation 4) as a mathematical representation of the electrode behavior.

$$E = E_0 + 2.3(RT/F)\,log\,aH^+ \qquad (4)$$

Where E is the cell potential in millivolts, E_0 is a calibration constant, R is the universal gas constant, T is the temperature in Kelvin, F is the Faraday constant, and aH^+ is the hydrogen-ion activity, which can be approximated by the H^+ concentration in most cases.

Manipulation of the Nernst equation generates the working equation used by pH analyzers: $E - E_0 = -slope\,(pH)$. The term $2.3(RT/F)$, then, represents the slope of a graph where E is plotted against $log\,aH^+$. The working equation allows pH analyzers to calculate the slope using two different buffer solutions, and then calculate E_0 (the zero offset). The ideal slope can be calculated using fundamental constants, but in practice, the slope is always lower than ideal. The ratio of actual slope to ideal slope is sometimes called the efficiency of the electrode. At 25°C, the ideal slope is 59.16 mV per pH unit, but many electrodes have slope values closer to 57 or 58 (Figure 2). This translates to electrode efficiencies of 96–98%.

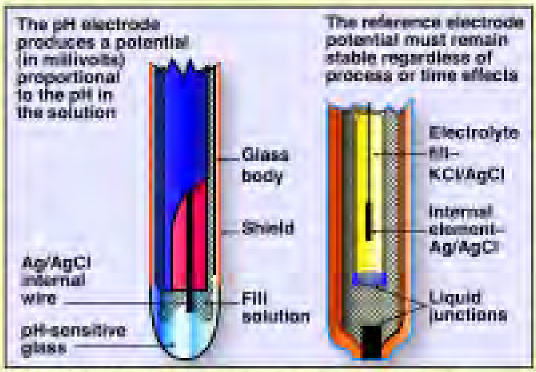

FIGURE 1. Most pH sensors contain these components

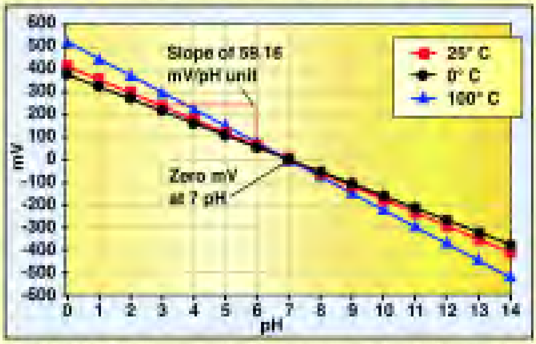

FIGURE 2. The theoretical millivolt response of a pH sensor is shown here

Temperature dependence

When measuring pH in the chemical process industries, accounting for temperature is important from several perspectives, including dependence on temperature of the slope of the measuring electrode, the effects of temperature on the reference point E_0, and on water dissociation.

The slope term of the Nernst equation contains the temperature, so the slope is proportional to temperature. The ideal slope varies from 54.2 mV per unit pH at 0°C to 74 mV per pH unit at 100°C. Most modern pH meters automatically compensate for the variation by simultaneously measuring process temperature and adjusting the pH reading accordingly.

Temperature effects on the reference E_0 can cause the calibration to shift. This influence is removed by using fill solutions inside the reference electrode with specific compositions that establish the zero point, and those at constant potentials (isopotential points) at a convenient pH value (usually 7). This means the sensor will read 0 mV at a pH of 7, and will continue to read the same if temperature is changed.

The dissociation constant also depends on temperature, because higher temperature causes more water molecules to dissociate, thus producing more H^+ and OH^- ions. The implication is that neutral pH varies from 7.45 near 0°C to 6.2 at 100°C.

The temperature behavior of non-neutral solutions can be complicated. In general, the pH values of acidic solutions change very little, while high-pH solutions take on lower pH values as temperatures rise. The net excess of OH^- ions in basic solutions causes the pH to drop at higher temperatures because of the large relative change in H^+ ions as the dissociation constant shifts. For example, a solution with pH 10 at 25°C has a pOH of 4 because the pK_w is 14. As the temperature rises close to 100°C, the pK_w decreases to around 12.4. Although equal amounts of H^+ and OH^- are formed, the H^+ concentration increases by a factor of 40, since there was only a small amount present before. So the pH of the solution shifts from 10 to 8.4, while the pOH changes very little.

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WHO'S WHO


Badami


Goodwin


York


Craighead


Jacobs

Harish Badami joins **Celanese Corp.** (Dallas, Tex.) as president of Celanese India, based in Mumbai.

Andy Goodwin is named president of the Bangkok-based Asian operations for **Pöyry Oyj** (Helsinki, Finland).

Bruce York joins **Endicott Biofuels LLC** (Houston, Tex.) as vice president of construction.

Oilfield service provider **Baker Hughes** (Houston, Tex.) names *Martin Craighead* president and CEO.

Grundfos Pumps (Olathe, Kan.) promotes *Greg Bretz* to director of regional program management.

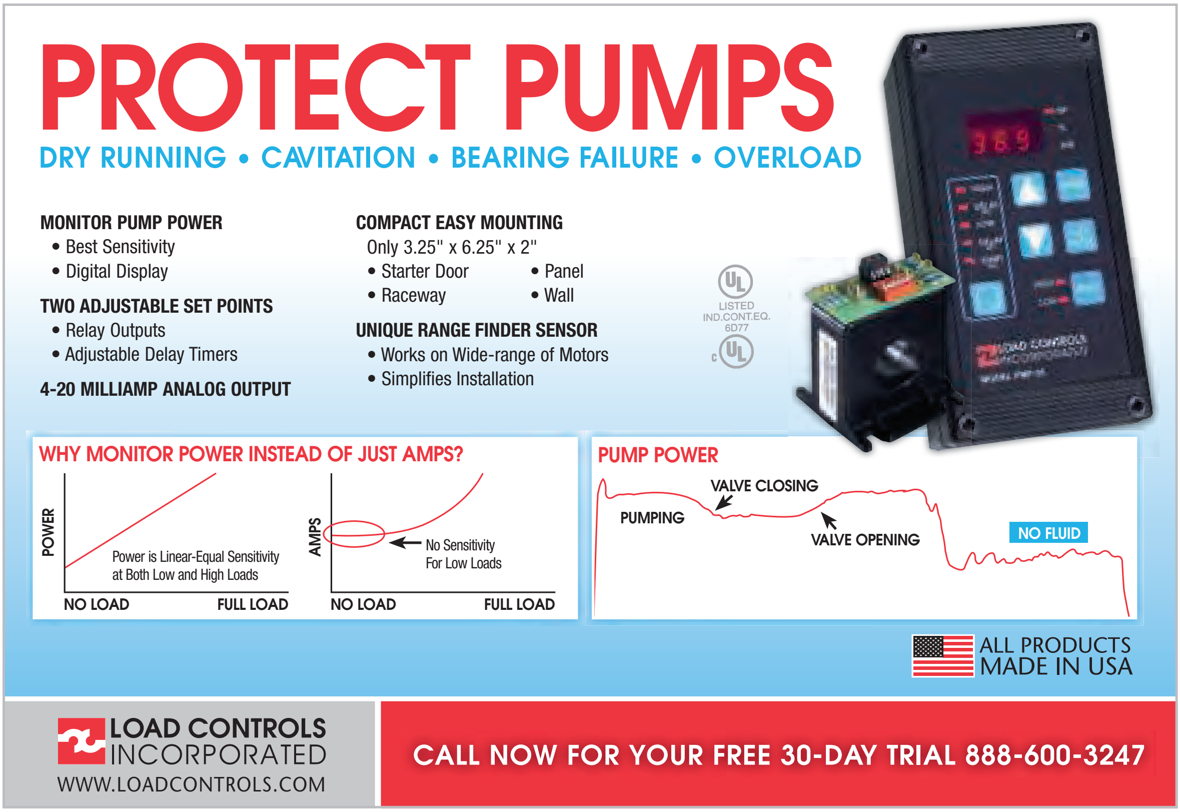
Mississippi Lime Co. (St. Louis, Mo.) welcomes *John Huhn* as vice president of strategy and corporate development.

Charkit Chemical Corp. (Norwalk, Conn.) names *Len Hyde* account representative for the Southwest U.S.

Trolex Ltd. (Cheshire, U.K.), a manufacturer of gas-detection and monitoring systems, announces the following: *Neil McDonald* is international sales director, *Qinglin Li* is business development manager (responsible for developing business in China), and *Tim Allard* is key accounts manager.

Mike Jacobs, president of **Applied Manufacturing Technologies** (Orion, Mich.), a supplier of plant automation design, engineering and consulting, has been re-elected to the Robotic Industries Assn.'s board of directors. ■

Suzanne Shelley



Effective Thermal Design Of Cooling Towers

A step-by-step approach to cooling-tower design, with an example calculation to make it clear

Jonny Goyal
Air Liquide Engineering and Construction, Lurgi India

NOMENCLATURE

t_2	Hot water temperature, °C
t_1	Cold water temperature, °C
t_{wb}	Wet-bulb temperature, °C
t_{db}	Dry-bulb temperature, °C
t_d	Dew point temperature, °C
h_a	Enthalpy of moist air, kJ/kg
h_1	Enthalpy of inlet air, kJ/kg
h_2	Enthalpy of exit air, kJ/kg
h'	Enthalpy of fin, kJ/kg
F	Flowrate, m^3/h
L	Mass flowrate of liquid, lb/h
G	Mass flowrate of gas, lb/h
Q	Heat load, kcal/h
Z	Altitude above sea level, m
p	Barometric pressure, kPa
p_{ws}	Saturation pressure of water vapor, kPa
p_w	Partial pressure of water vapor, kPa
v	Specific volume, m^3/kg
W	Humidity ratio, kg water/kg air
W_s	Humidity ratio at saturation air, kg water/kg moist air
φ	Relative humidity (RH), %
C	Constant related to cooling tower design
m	Slope of tower characteristic curve

Various misconceptions arise when it comes to the thermal design of cooling towers. Sometimes related parameters, such as range, approach, effectiveness, liquid-to-gas ratio (L/G), wet-bulb temperature, cooling water temperature, relative humidity, number of transfer units (NTU) and other terms create a confusion for the designer in effectively sizing, selecting and evaluating a particular cooling tower. This leads to inadequate design.

The objective of this article is to present a stepwise understanding of how to calculate the NTU for a cooling tower, and thus to understand the basis of thermal design of counterflow cooling towers for optimizing cost and performance.

Definitions

First, let's look at some of the basic terms and briefly describe their significance and role in cooling tower design and performance.

Dry-bulb temperature. Dry-bulb temperature (t_{db}) — usually referred to as the air temperature — is the property of air that is most commonly used. When people refer to the temperature of the air, they are normally referring to its dry-bulb temperature. The dry-bulb temperature is an indicator of heat content and is shown along the bottom axis of a psychometric chart. The vertical lines extending upward from this axis are constant-temperature lines.

Wet-bulb temperature. Wet-bulb temperature (t_{wb}) is the reading when the bulb of a thermometer is covered with a wet cloth, and the instrument is whirled around in a sling. The wet-bulb temperature is the lowest temperature that can be reached by evaporation of water only.

Relative humidity (RH). RH is the ratio of the partial pressure of water vapor in air over the saturation vapor pressure at a given temperature. When the relative humidity is 100%, the air is saturated and therefore, water will not evaporate further. Therefore, when the RH is 100% the wet-bulb temperature is the same as the dry-bulb temperature, because the water cannot evaporate any more.

Range. The range is the difference in temperature of inlet hot water (t_2) and outlet cold water (t_1), $t_2 - t_1$. A high cooling-tower range means that the cooling tower has been able to reduce the water temperature effectively.

Approach. The approach is the difference in temperature of outlet cold water and ambient wet-bulb temperature, $t_1 - t_w$. The lower the approach, the better the cooling tower performance. Although both range and approach should be monitored, the approach is a better indicator of cooling tower performance.

Cooling tower capability. The capability of the cooling tower is a measure of how close the tower can bring the hot water temperature to the wet-bulb temperature of the entering air. A larger cooling tower (that is, more air or more fill) will produce a closer approach (colder outlet water) for a given heat load, flowrate and entering air condition. The lower the wet-bulb temperature, which indicates either cool air, low humidity or a combination of the two, the lower the cooling tower can cool the water. Capability tests are conducted per the ATC-105 Code of the Cooling Tower Institute (CTI; Houston; www.cti.org).

The thermal performance of the cooling tower is thus affected by the entering wet-bulb temperature. The entering air dry-bulb temperature has an insignificant effect on thermal performance.

Effectiveness. A cooling tower's effectiveness is quantified by the ratio of the actual range to the ideal range, that is, the difference between cooling water inlet temperature and ambient wet-bulb temperature. It is defined in terms of percentage.

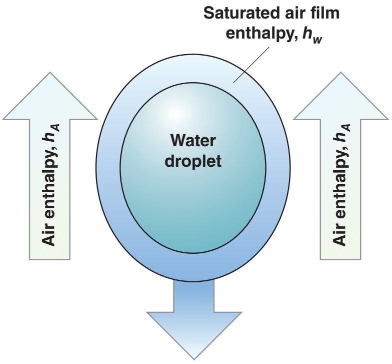


FIGURE 1. The Merkel equation is derived by considering a falling water droplet surrounded by saturated air

$$\text{Effectiveness} = \frac{\text{Range}}{\text{Range} + \text{Approach}} \times 100\% \qquad (1)$$

Liquid-to-gas ratio (L/G). The L/G ratio of a cooling tower is the ratio of the liquid (water) mass flowrate (L) to gas (air) mass flowrate (G). Cooling towers have certain design values, but seasonal variations require adjustment and tuning of water and air flowrates to get the best cooling tower effectiveness.

Number of transfer units (NTU). Also called the tower coefficient, the NTU is a numerical value that results from theoretical calculations based on a set of performance characteristics. The value of NTU is also representative of the degree of difficulty for the cooling process. The NTU corresponding to a set of hypothetical conditions is called the *required coefficient* and is an evaluation of the problem. The same calculations applied to a set of test conditions is called the *available coefficient* of the tower involved. The available coefficient is not a constant but varies with operating conditions. The operating characteristic of a cooling tower is developed from an empirical correlation that shows how the available coefficient varies with operating conditions.

Cooling capacity. The cooling capacity of a tower is the heat rejected [kcal/h or TR (refrigeration tons; 1 TR = 12,000 Btu/h = 3,025.9 kcal/h)], and is determined by the product of mass flowrate of water, times the specific heat times the temperature difference.

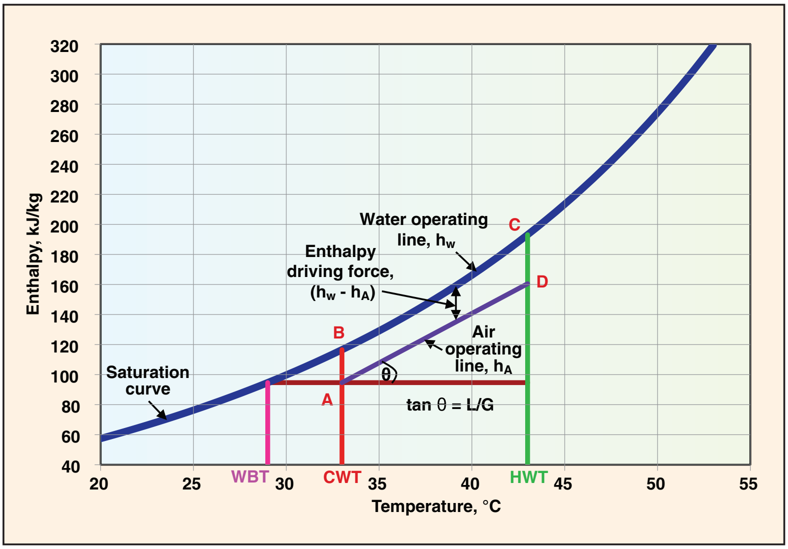


FIGURE 2. This plot, known as the driving force diagram, shows the enthalpy versus temperature for water and air

Theory — the Merkel equation

In a cooling tower operating in counter current flow, there are two basic principles involved for removing heat by the cooling water:

1. sensible heat transfer due to a difference in temperature levels
2. latent heat equivalent of the mass transfer resulting from the evaporation of a portion of the circulating water

Merkel developed the basic equation based on the above principles. The Merkel model is universally accepted for designing and rating of counterflow cooling towers. The model is based on a drop of water falling through an upstream flow of unsaturated air at a wet-bulb temperature of t_{wb} with enthalpy h_A (Figure 1), in a counterflow cooling tower. The drop of water is assumed to be surrounded by a film of saturated air at the water temperature WT with saturation enthalpy h_W. As the drop travels downward, heat and mass transfer takes place from the interface air film to the upstream air, thereby cooling the water from hot temperature to a cold temperature.

The main assumptions of Merkel theory are the following:

1. The saturated air film is at the temperature of the bulk water.
2. The saturated air film offers no resistance to heat transfer.
3. The vapor content of the air is proportional to the partial pressure of the water vapor.
4. The heat transferred from the air to the film by convection is proportional to the heat transferred from the film to the ambient air by evaporation.
5. The specific heat of the air-water vapor mixture and the heat of vaporization are constant.
6. The loss of water by evaporation is neglected.
7. The force driving heat transfer is the differential enthalpy between the saturated and bulk air.

This cooling process can best be explained on a psychometric chart, which plots enthalpy versus temperature. The process is illustrated in the so-called driving-force diagram shown in Figure 2. The air film is represented by the water operating line on the saturation curve. The main air is represented by the air operating line, the slope of which is the ratio of liquid (water) to air (L/G). The cooling characteristic, a degree of difficulty to cooling is represented by the Merkel equation:

$$KaV/L = \int_{CWT}^{HWT} \frac{dT}{h_W - h_A} \qquad (2)$$

Where:

K = overall enthalpy transfer coefficient, lb/h-ft^2
a = Surface area per unit tower volume, ft^2/ft^3
V = Effective tower volume, ft^3
L = Water mass flowrate, lb/h

Equation 2 basically says that at any point in the tower, heat and water vapor are transferred into the air due (approximately) to the difference in

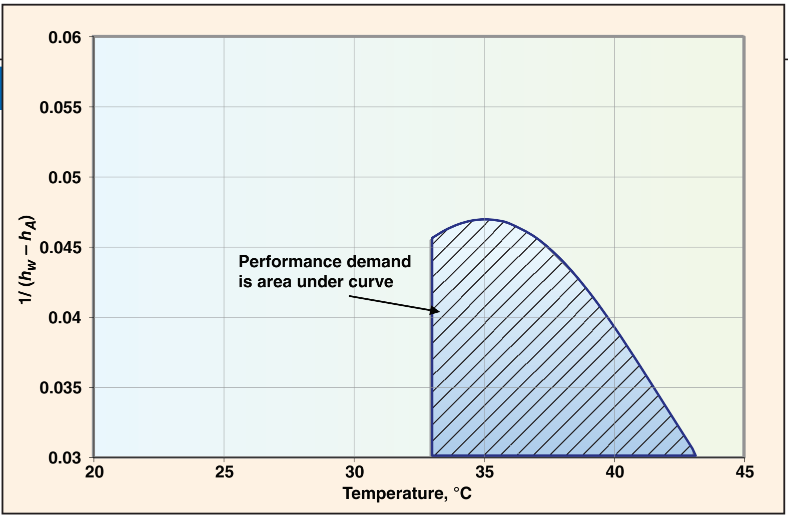


FIGURE 3. Solving the Merkel equation (Equation 2), is usually done graphically, where the integral is equal to the area under the curve

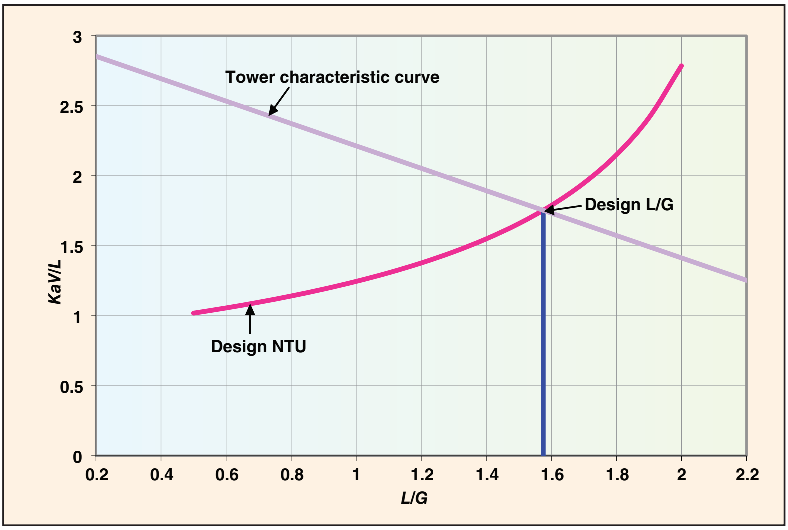


FIGURE 4. The intersection of the tower characteristic curve and the design NTU curve gives the design *L/G* ratio

the enthalpy of the air at the surface of the water and the main stream of the air. Thus, the driving force at any point is the vertical distance between the two operating lines. And therefore, the performance demanded from the cooling tower is the inverse of this difference. The solution of the Merkel equation can be represented by the performance demand diagram shown in Figure 3. The KaV/L value is equal to the area under the curve, and represents the sum of NTUs defined for a cooling tower range.

An increase in the entering t_{wb} moves the air operating line towards the right and upward to establish equilibrium. Both the cold water temperature (CWT) and hot water temperature (HWT) increases, while the approach decreases. The curvature of the saturation line is such that the approach decreases at a progressively slower rate as the t_{wb} increases. An increase in the heat load increases the cooling ranges and increases the length of the air operating line. To maintain equilibrium, the line shifts to the right increasing the HWT, CWT, and approach. The increase causes the hot water temperature to increase considerably faster than does the cold water temperature. In both these cases, the KaV/L should remain constant. However, a change in L/G will change the KaV/L value.

Cooling tower design

On the basis of the above discussion, it is clear that there are five parameters that, in combination, dictate and define the performance of a cooling tower, namely:

1. Hot water temperature, HWT
2. Cold water temperature, CWT
3. Wet bulb temperature, t_{wb}
4. Water mass flowrate, L
5. Air mass flowrate, G

The first four parameters are determined by the user of the cooling tower. It is the fifth quantity, G, that is selected by the designer of the cooling tower. Once these five quantities are available, the tower characteristic (KaV/L), can be calculated through the Merkel equation.

The first step in designing a cooling tower is the generation of a demand curve. In this curve, the KaV/L values are plotted against varying L/G ratios. The next step is to superimpose fill-characteristic curves and demand curves. The Cooling Technology Institute has tested a variety of fill configurations and generated fill characteristic curves for each type; CTI's Technical Paper TP88_05 can be referred to in this regard.

Cooling tower design is basically an iterative process. The factors that effect the selection of design L/G and consequently the fill height are: cell dimensions, water loading, air velocities across various cooling tower sections and pressure drops, and fan selection.

The classical method of thermal rating of cooling towers is to estimate the ratio of liquid to gas first and then find the proper tower volume by the means of trial and error using the tower performance curve. The L/G is the most important factor in designing the cooling tower and related to the construction and operating cost of cooling tower.

Finally we can summarize the importance of the L/G ratio with the following points.

A high L/G ratio means:

- More water to less air
- Air is more saturated — driving force is reduced
- More residence time of water needed
- Less cooling in given time
- Increase in required fan power
- Decrease in height of tower
- Low evaporation loss (under same water flowrate)

An example makes it clear

As an example, let us design a cooling tower with the following data:

TABLE 1.

Water Temperature, t		Water Vapor Saturation Pressure, p_{ws}	Partial Press. of H_2O Vapor, p_w	Humidity Ratio, W	Enthalpy of Film, h'	Enthalpy of Air, h_a	Enthalpy Difference, $(h'-h_a)$	$1/(h'-h_a)$	Δt	NTU	ΣNTU	Cumulative Cooling Range
°C	K	kPa	kPa	kg water/ kg dry air	kJ/kg	kJ/kg	kJ/kg	kg/kJ				
33	306.15	5.0343	5.0343	0.0326	116.5686	94.6668	21.9018	0.0457				
33.5	306.65	5.1774	5.1774	0.0335	119.5982	97.9585	21.6397	0.0462	0.5	0.096	0.096	0.5
34	307.15	5.3239	5.3239	0.0345	122.6986	101.2503	21.4483	0.0466	0.5	0.097	0.193	1
34.5	307.65	5.4740	5.4740	0.0356	125.8718	104.5420	21.3298	0.0469	0.5	0.098	0.291	1.5
35	308.15	5.6278	5.6278	0.0366	129.1197	107.8338	21.2859	0.0470	0.5	0.098	0.389	2
35.5	308.65	5.7853	5.7853	0.0377	132.4444	111.1255	21.3189	0.0469	0.5	0.098	0.487	2.5
36	309.15	5.9466	5.9466	0.0388	135.8480	114.4173	21.4307	0.0467	0.5	0.098	0.585	3
37	310.15	6.2810	6.2810	0.0412	142.9006	121.0008	21.8999	0.0457	1.0	0.193	0.778	4
38	311.15	6.6315	6.6315	0.0436	150.2958	127.5843	22.7116	0.0440	1.0	0.187	0.965	5
39	312.15	6.9987	6.9987	0.0462	158.0530	134.1678	23.8852	0.0419	1.0	0.180	1.145	6
40	313.15	7.3835	7.3835	0.0489	166.1928	140.7513	25.4415	0.0393	1.0	0.170	1.314	7
41	314.15	7.7863	7.7863	0.0518	174.7371	147.3348	27.4023	0.0365	1.0	0.158	1.473	8
42	315.15	8.2080	8.2080	0.0549	183.7094	153.9183	29.7911	0.0336	1.0	0.146	1.619	9
43	316.15	8.6492	8.6492	0.0581	193.1348	160.5018	32.6330	0.0306	1.0	0.134	1.753	10
									ΣNTU	1.75334674		

Capacity (F): 3,000 m^3/h
Wet bulb temperature (t_{wb}): 29°C
Relative humidity (φ): 92%
Cooling water inlet (t_2): 43°C
Cooling water outlet (t_1): 33°C
Altitude (Z): 10 m

Step I. This step involves heat load calculations as follows:

1. Range = $(t_2 - t_1) = 43 - 33 = 10$°C
2. Approach = $(t_1 - t_{wb}) = 33 - 29 = 4$°C
3. Heat load, $Q = mC_p(t_2 - t_1)$
   $= 998.13 \times F \times$ Range
   $= 998.13 \times 3{,}000 \times 10$
   $= 29{,}943{,}900$ kcal/h

Step II. This step involves total psychometric calculations as follows:

1. Barometric pressure (p) at the given altitude (Z) is calculated by using the following equation:

$$p = 101.325(1 - 2.25577 \times 10^{-5} Z)^{5.2559} \quad (3)$$

For an altitute of 10 m, this becomes $p = 101.2$ kPa

2. Assume a dry bulb temperature of say, $t_{db} = 32$°C
3. Calculate water vapor saturation pressure (p_{ws}) at the assumed t_{db} for the temperature range of 0 to 200°C using the equation:

$$\ln(p_{ws}) = \left[C_1 T^{-1} + C_2 + C_3 T + C_4 T^2 + C_5 T^3 + C_6 \ln(T)\right] \div 1{,}000 \quad (4)$$

Where:
$C_1 = -5.8002206 \times 10^3$
$C_2 = 1.3914993 \times 10^0$
$C_3 = -4.8640239 \times 10^{-2}$
$C_4 = 4.1764768 \times 10^{-5}$
$C_5 = -1.4452093 \times 10^{-8}$
$C_6 = 6.5459673 \times 10^0$

and T represents the dry bulb temperature in Kelvin. This results in the value:
$p_{ws} = 4.7585$ kPa

4. The partial pressure of water (p_w) at given relative humidity is found using the following equation:

$$p_w = p_{ws} \times \varphi \quad (5)$$

   $p_w = 4.3779$ kPa

5. The partial pressure (p_{ws}) is again calculated using Equation 4. This time T represents the wet bulb temperature in Kelvin, which calculates to:
$p_{ws} = 4.0083$ kPa

6. Using p_{ws} calculated in Step 5 we recalculate t_{wb} using the Carrier equation:

$$p_w = p_{ws} - \left\{ \frac{1.8 \times (p - p_{ws}) \times (t_{db} - t_{wb})}{\left[2{,}800 - 1.3 \times (1.8 t_{wb} + 32)\right]} \right\} \quad (6)$$

   which gives the result:
   $t_{wb} = 37.7$°C

7. This step is an iterative process, whereby the assumed value of t_{db} in Step 2 is varied in such a way that the calculated t_{wb} in Step 6 equals the actual (real) t_{wb}.
8. After a number of iterations, the calculated t_{db} value converges to 30.12°C.

Step III. This step involves the calculation of the inlet air enthalpy (h_1) as follows:

1. The humidity ratio (W) for dry air is calculated using the following equation:

$$W = \frac{0.62198 \times p_w}{p - p_w} \quad (7)$$

$W = 0.02515$ kg water/kg dry air

2. The specific volume (v) for dry air is calculated using the following equation:

$$v = \frac{0.2871 \times (t_{db} + 273.15) \times (1 + 1.6078W)}{p} \quad (8)$$

   $v = 0.89511$ m^3/kg, dry air

3. Calculate the enthalpy of inlet air (h_1) using the following equation:

$$h_1 = 1.006 t_{db} + W(2{,}501 + 1.86 t_{db}) \quad (9)$$

   $h_1 = 94.750$ kJ/kg

4. Calculate the humidity ratio at saturation (W_s) for wet air using same Equation 7. Here we now use p_{ws}:

   $W_s = 0.02743$ kg water/kg moist air

TABLE 2. TYPICAL THERMAL CALCULATIONS OF COUNTER-FLOW COOLING TOWER

Flowrate	3,000	m^3/h	Hot water temperature	43	°C
Wet-bulb temperature	29	°C	Cold water temperature	33	°C
Approach	4	°C	Range	10	°C
Assumed dry-bulb temperature	30.12	°C	Heat load	29943888.32	kcal/h
Assumed L/G	1.575		Barometric pressure (p)	101.2	kPa
No. of cells	3				
Cell length	14	m	Water vapor saturation pressure (p_{ws}), at t_{db}	4.2754	kPa
Cell width	14	m	Partial pressure of water vapor (p_w)	3.9333	kPa
Air inlet height	5.5	m	Water vapor saturation pressure (p_{ws}), at t_{wb}	4.0083	kPa
Design RH	92%		Recalculating t_{wb}	29.0	°C
Density of water	1,000	kg/m^3	Difference	0.0	°C
Altitude	10	m	Dew point temperature (t_d) of moist air (for temperature between 0 and 93°C)	28.66	°C

INLET AIR PROPERTIES		
Inlet t_{wb}	29	°C
RH	92%	
Inlet t_{db} at above RH	30.12	°C
Humidity ratio (W)	0.0252	kg water/kg dry air
Specific volume (v)	0.8951	m^3/kg, dry air
Density	1.1172	kg/m^3, dry air
Humidity ratio at saturation (W_s)	0.0274	kg water/kg moist air
Specific volume, (v) at saturation	0.8983	m^3/kg, moist air
Density	1.1133	kg/m^3, moist air
Enthalpy of moist air	94.5702	kJ/kg
Humidity Ratio at t_{wb}	0.0256	kg water/kg moist air
Enthalpy at t_{wb}	94.6668	kJ/kg

AIR FLOW		
Average density dry	1.0827	kg/m^3
Average density wet	1.0801	kg/m^3
Air flowrate at fin	162.90	m^3/s, per cell
Air flow at inlet, at rain zone	157.87	m^3/s, per cell
Air flowrate at fan	168.26	m^3/s, per cell

EXIT AIR PROPERTIES		
Enthalpy	160.50	kJ/kg
Exit air temperature (t_{db})	39.55	°C
Water vapor saturation pressure (p_{ws}), at t_{db}	7.2097	kPa
Put some value of exit RH (Assuming it is in between 97 and 99%)	98.5	%
Partial pressure of water vapor (p_w)	7.1016	kPa
Humidity ratio (W) at above t_{db}	0.04692	kg water/kg dry air
Enthalpy of exit air	160.50	kJ/kg
Difference	0.00	
Exit t_{wb}	39.31	°C
Water vapor saturation pressure (p_{ws}) at exit t_{wb}	7.1170	kPa
Recalculating t_{wb}	39.31	°C
Difference	0.00	
Dew point temperature (t_d) of moist air (for temperature between 0 and 93°C)	39.24	°C
Specific volume (v)	0.9540	m^3/kg, dry air
Density	1.0482	kg/m^3, dry air
Humidity ratio (W_s) at saturation	0.0477	kg water/kg moist air
Specific volume (v) at saturation	0.9551	m^3/kg, moist air
Density	1.0470	kg/m^3, moist air
KaV/L	1.7533	
Constant related to tower design (C)	2.522	

5. Calculate the specific volume (v) for wet air using Equation 8 with W_s.

   v = 0.89827 m³/kg moist air

Step IV. This step involves the calculation of the exit air properties, as follows:

1. Assume some value of the L/G ratio, say 1.575, and calculate h_2 for exit air using the following equation:

$$h_2 = h_1 + \left[(L/G) \times Range \times 4.186\right] \quad \textbf{(10)}$$

   h_2 = 160.50 kJ/kg

2. Assume that the exit air has a relative humidity of 97–99% (design RH at the outlet), and also assume some value of exit air dry-bulb temperature.
3. Use the same partial pressure and humidity equations as discussed in Step II and Step III, to calculate the enthalpy of exit air at these assumed values in Point 2 above. At an assumed RH of 98.5% and exit air t_{db} of 37°C, we recalculate

   h_2 = 141.18 kJ/kg

4. This is again an iterative process. Next, assume the value of exit air t_{db} in Point 2 (at same relative humidity) in such a way that the calculated h_2 in Point 3 equals the h_2 calculated in Point 1.
5. After a number of iterations, the calculated exit air t_{db} value converge to 39.56°C.
6. Now that the dry-bulb temperature and RH are known values, recalculate the wet-bulb temperature using Equation 6.

   t_{wb} = 39.31°C

7. Calculate the dry and wet specific volume of exit air using Equation 8. Also calculate the density of dry air and wet air (for inlet and exit).
   v = 0.8540 m³/kg, dry air
   v = 0.8551 m³/kg, moist air
   Average density dry = 1.0827 kg/m³
   Average density wet = 1.0801 kg/m³

Step V. This step is to help draw the driving force diagram as follows:

1. Take different temperature ranges (covering cooling water inlet and outlet temperature) and calculate the enthalpy of air using Equation 9 and psychometric calculations discussed above. Plot the air saturation curve (enthalpy versus temperature) as shown in Figure 2.
2. Take cooling water outlet temperature and calculate the enthalpy of film (h') using Equation 9. Here the partial pressure, saturation pressure and humidity ratio are calculated for the corresponding temperature taken (Table 1).

h' at 33°C = 116.569 kJ/kg

This enthalpy specifies Point B on the graph of Figure 2, and is the starting point of the water operating line.

3. Similarly calculate enthalpy of air h_a at wet-bulb temperature (Table 1).

h_a = 94.667 kJ/kg

This enthalpy specifies Point A on the graph of Figure 2 and is the starting point of the air operating line.

4. Take incremental change in temperature (say 0.5 or 1.0) up to the hot water temperature and calculate the h' and h_a. The ending points are shown as C and D in Figure 2 on the water operating and air operating line respectively.
5. The difference between h' and h_a will give you the enthalpy driving force for incremental change in temperature.
6. Take the inverse of enthalpy difference in each incremental step (Table 1).
7. Calculate NTU = 4.18 × Δt × (Average of incremental increase in inverse of enthalpy difference),

Or for 0.5°C increment in temperature, calculated NTU = 0.096

8. Similarly, calculate the NTU for each step and add to get the total NTU for the particular assumed L/G ratio (Table 1). Or, for an assumed L/G of 1.575, Total NTU = 1.7533 — this is KaV/L.
9. Now to plot the tower characteristic curve, first we vary the L/G ratio and repeat all calculations discussed above to generate the data for various NTU to plot. The curve represents "Design NTU" on the graph, shown in Figure 4.
10. Take the design L/G ratio and plot the tower characteristic curve by assuming the slope of the line (m), which usually varies between –0.5 to –0.8. One can also consult with vendors for this value as it also depends on the type of fill used.
11. Calculate the value of the constant C, related to cooling tower design using equation:

$$NTU = C \times (L/G)^m$$

$$C = 2.522$$

The significance of these calculations is that now we can directly calculate the cooling tower characteristic by using our equations and can compare with the vendor data if the provided height of the cell is adequate to meet the calculated NTU. Obviously the height also depends on the type of packing, but along with vendor input we can create a complete economical design of our cooling tower. Further, we can develop a calculation sheet in Microsoft Excel, which gives all the results of the psychometric chart as well as the cooling tower design. Typical thermal calculations for a counter-flow cooling tower can be seen in Table 2. ■

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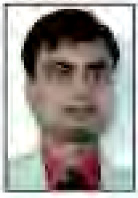

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Vessel Sizing and Level Instrumentation

When sizing process vessels, it is important to consider instrumentation. Consistent, safe and reliable results depend on it

Chad Schaffer
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Level measurement and control in process vessels can be one of the most challenging applications in which to obtain consistently safe and reliable results. Part of this difficulty may be due to the fact that the sizing of the vessel and selection and installation of the level instruments are frequently performed by different people at different times, rather than evaluated holistically as a system.

Level instruments are installed on vessels to provide local indication of the liquid level, automatic level control and alarms to indicate upset conditions so that an operator can respond. They are also used to automatically bring a process to a safe state when safe operating limits are reached. The level-instrument technologies used and how they are installed can adversely affect the usable vessel volume by reducing the span that is visible to the operator and the control system.

This article presents best practices in vessel sizing and level-instrument selection and installation. Practical examples demonstrate the recommended holistic approach.

Vessel sizing

While various methods exist in the chemical process industries (CPI) for sizing vessels, two methods find widespread use.

Method 1. One of the most common methods to size vessels is presented in "Chemical Process Equipment – Selection and Design" [1]. This method states that the vessel volume is calculated in terms of the number of minutes of flow (or the residence time) on a half-full basis, and that five to ten minutes is adequate for most applications. Fired-heater feed surge drums and the liquid sumps of compressor-suction knockout drums are specific exceptions where more residence time is required. Method 1 is commonly used because the only information needed to size the vessel is the volumetric flowrate and knowledge of the equipment being fed by the vessel. Figure 1 depicts an example of vessel sizing on this basis, using a 1,000 gal/min flowrate and a 5-min residence time.

Method 2 and variants. Another method used to size vessels is presented in "Distillation Operation" [2]. This method is presented specifically for distillation reflux drums, but is easily adapted for sizing other types of process vessels by adjusting the residence time requirement to suit the process intent. Rather than assuming the vessel is operating at half-full, it recommends sizing based on the residence time between the high- and low-level operating limits. Method 2 is slightly more complex than Method 1 because it also requires knowledge of the vessel operating limits in order to determine the total vessel volume. Figure 2 depicts an example of vessel sizing on this basis, again using a 1,000 gal/min flowrate and a 5-min residence time.

Other sizing methodologies, which are variants of the second method, may take into account specific hazards that can exist if a vessel runs empty or is overfilled. For example, if a hazard is created for downstream equipment when a vessel runs empty — such as due to vapor blow-through or running the tubes in fired equipment dry — then it may be appropriate to also consider the residence time from the low-level operating limit to empty. Conversely, if a hazard is created when a vessel overfills — such as a compressor suction drum — then considering the residence time from the high-level operating limit to the overfill or liquid-carryover condition can be suitable. In either case, the purpose of the vessel and its operating

FIGURE 1. In Method 1, vessels are sized based on flowrates and residence time

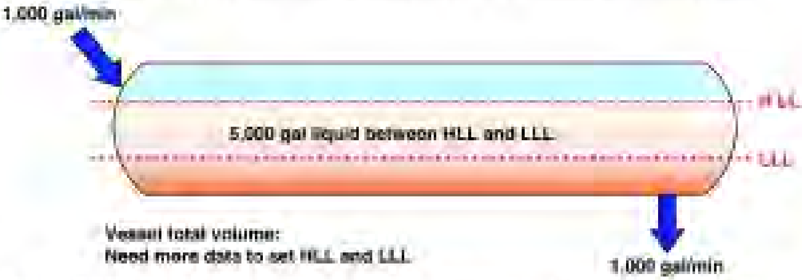

FIGURE 2. Method 2 recommends sizing based on residence time between the high-level limit (HLL) and low-level limit (LLL) of operation

FIGURE 3. A magnetic level gage can address the limitations associated with simple gage glasses

limits must be thoroughly understood to use these methods.

Vessel sizing best practices. When vessels are sized during conceptual design for the purpose of developing a budgetary or funding-approval estimate, there is generally insufficient information available to define the operating limits. Since cost estimates are typically on the order of ±25 to 50% during these early design phases, Method 1 is suitably accurate and is easy to apply.

When making final vessel-sizing calculations during the definitive estimate phase (typically ±10%), additional information should be taken into consideration in order to ensure that the usable volume is sufficient to perform the vessel's function. In addition to knowledge of the operating limits required by Method 2, this typically involves information that may not be readily available to the engineers responsible for sizing the vessel, and so often requires involvement of a design team to determine the following:

- What level-instrument technologies will be used
- How the level instruments will be installed
- Alarms and the time required for an operator to respond appropriately before a hazardous situation occurs or an automated system takes action to make the process safe

In many cases these two methods will yield similar results, but spending a small amount of time optimizing the design with some additional knowledge can pay big dividends in how much operating range is actually available in the vessel.

Local level instruments

Level instruments intended for local indication allow visual confirmation of the level for redundancy in operation, as well as for calibration of level transmitters and testing of level switches. Two frequently used types are discussed below.

Gage glasses. A single-section gage glass is a simple and intuitive device for viewing liquid level. One limitation of gage glasses relative to the usable volume of a vessel is that they usually extend 3 in. or more at both the top and the bottom (6 in. total) of the gage, which is not covered by the glass window and therefore should not be considered part of the usable, vessel operating range. Because of the risk of breakage and loss of containment of the process media, multiple overlapping gages (usually of 5-ft or shorter lengths) are often required for taller vessels, which may require more vessel platforms for access to the instrument block, vent and drain valves.

Magnetic level gages. Figure 3 depicts a magnetic level gage. A magnetic float is installed inside the chamber, and the indicators change the displayed color as the magnetic field of the float travels. This type of level instrument addresses the limitations associated with gage glasses because there is no loss of visible range between the connection points. Magnetic level gages can measure the entire span.

Glass breakage is not a concern with magnetic level gages, so measuring lengths of ten feet or sometimes more is easily handled in a single gage, which can mean fewer vessel platforms required for access to valves.

Additional installation considerations to be aware of are that magnetic level gages require significant clear space below the bottom connection for float removal, and are physically larger and heavier than a gage glass and so may require larger diameter or reinforced process connections to support their weight.

Transmitters and switches

Level transmitters and switches provide a method for the level in the vessel to be read remotely. They are used to provide automatic level control and alarms to indicate upset conditions and trigger operator response. They can also be used to automatically bring the process to a safe state if the operating limits are reached. Many transmitter and switch technologies are available, and no single technology will suit all applications.

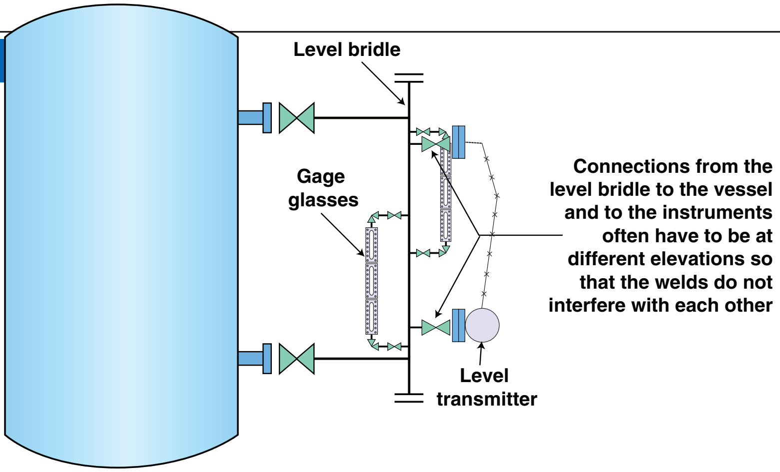

FIGURE 4. This level bridle with gage glasses and level transmitter on a vertical vessel depicts a common (but generally not recommended) installation practice

The measuring range of a transmitter is typically specified to be the same as or slightly less than (usually 4–6 in.) the measuring range of the local level gage. This allows the output of the transmitter to be directly compared to the visual indicator to check its calibration and ensure it is functioning properly. The transmitter range should rarely exceed the gage visible range, since this makes verifying the high and low limits of the transmitter in situ difficult, if not impossible. Conversely, any vessel volume that is viewable by the local gage but not measurable by the transmitter is generally not available for use in the operating range.

The working operating range of the liquid level should not extend past the alarm points, and by definition cannot extend past the shutdown points.

Typically the vessel percent full as read by the level instrument correlates to percent of level instrument span, and does not equate to percent of actual vessel volume. A vessel strapping table should be developed and used to convert percent level readings to percent of vessel volume. Most modern control systems can be programmed with an equation to convert automatically, if desired.

The alarm and shutdown setpoints for high level generally should be no greater than 90% of the transmitter span. Similarly, the alarm and shutdown setpoints for low level should be no less than 10% of the transmitter span. The reason for this is to ensure that these critical points can be read by the transmitter under all operating conditions.

For example, when using a differential-pressure-style level transmitter, a 10% decrease in the density of the process fluid — such as might occur in vessels containing fluid mixtures of varying composition or temperature — can result in the vessel being completely full (filled above the top transmitter connection), but the transmitter may indicate only 90% output. This is because the weight of the liquid column between the transmitter connections is less than that for which the transmitter was designed, and the liquid above the top connection exerts its weight on both the top and the bottom connections equally, so it does not create a differential pressure to be measured. If the alarm or shutdown setpoint is greater than 90%, the transmitter may be incapable of reading it during process upset conditions when temperature or composition changes affect the liquid density.

The selection of the level technology to be used should include consideration of how the specific physical property of the liquid being measured may change during normal operating or process upset conditions. If the liquid density can change significantly, then differential-pressure-style measurements may not be the best choice. If the electrical conductivity of the liquid can vary, or is extremely low, then technologies that rely on this property, such as radar or capacitance, may not be suitable.

Any shutdown setpoint should generally be preceded by an alarm to alert the operator to take action to bring the process back into control and prevent safe operating limits from being exceeded. The difference between the alarm and shutdown setpoints needs to be adequate for the operator to have response time to identify that an issue exists, then diagnose and correct the problem. The alarm and shutdown setpoints also need to be sufficiently far away from the normal operating level that minor process upsets do not create nuisance alarms.

To ensure that the level-instrument design and the vessel process design are consistent with each other, the drum levels corresponding to low and high alarms and shutdown setpoints should be indicated on the process and instrumentation diagram (P&ID).

Level bridles

Level bridles are often used as a means to mount multiple level instruments to a single pair of vessel process connections, rather than providing each level instrument with independent connections to the vessel. This is sometimes unavoidable and necessary, especially with existing pressure vessels or high-pressure, thick-walled vessels where the addition of nozzles cannot be cost justified.

Mounting instruments to a level bridle generally requires that the elevations of the process connections (for each instrument to the bridle) be staggered to avoid interference with each other, and this results in a loss of usable vessel volume as the instrument span is narrowed. This consequence can be avoided if each instrument has its own connections to the vessel.

In addition, avoiding the use of level bridles will reduce the total number of welds required, generally reduce the number of valves required to install all the instruments, and will virtually eliminate an interface between the process control and piping design disciplines. All of these factors reduce the complexity and cost of the level-instrument installation.

Level instrument best practices

In order to maximize the volume of a vessel that is usable and within the measurable and controllable limits of the instrumentation, take the steps outlined here. Each of these three steps will add approximately 6 in. to the usable operating range of the liquid, or about 18 in. total.

1. Use magnetic level gages instead of

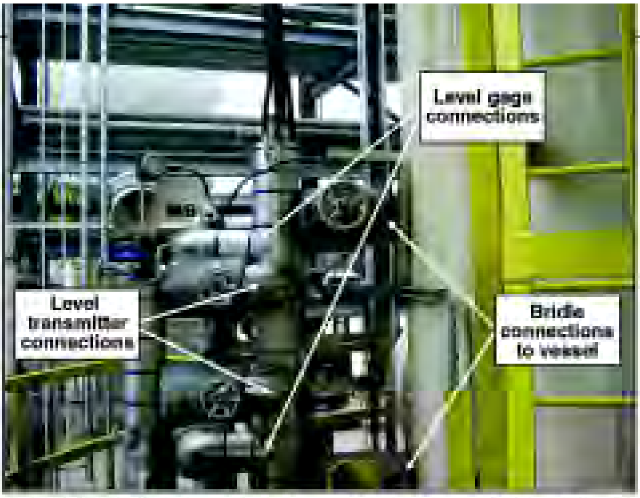

FIGURE 5. This installation is similar to that shown in Figure 4, with a staggering of the instrument connection elevations

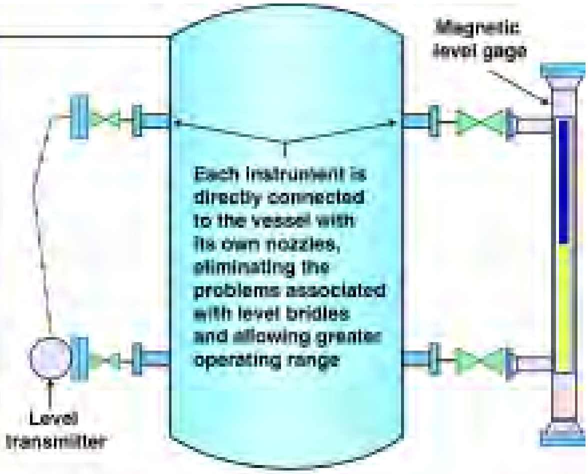

FIGURE 6. A best practice is to use a single magnetic level gage and a level transmitter

gage glasses where the technology is suitable for the application.

2. Install the level transmitters to measure the entire visible range of the local gage instead of covering a narrower range.
3. Mount each instrument directly to the vessel with its own process connections rather than off a level bridle.

The smaller the size of the vessel, the greater percentage of the total volume these 18 in. to be gained represent, so while important in all cases these best practices are most critical on horizontal vessels and water boots and other vertical vessels with a short sidewall.

Practical examples

Common practice. Figure 4 depicts a situation where a vertical vessel with dimensions of 8-ft. inside diameter (I.D.) by 12-ft tangent-to-tangent height (nominal volume 5,000 gal including the bottom head) is equipped with a level instrument setup that is very commonly encountered in industry. It has a side-mounted level bridle, two overlapping gage glasses, and a level transmitter. Walk around your facility and you'll probably find a few installations set up just like this. See Figure 5 for a similar example of what you may find.

The vessel was sized on the basis of Method 1. At 50% full, the vessel is designed for 5 min of residence time with 500 gal/min of liquid flow. Now evaluate how the level instruments are installed and what effect this has on the usable volume of the vessel.

The process connections for the level instruments are often approximately 12 in. away from the tangent lines, so in this case with the vessel height of 12 ft, the nozzles would be about 10 ft apart.

By mounting the gage glasses to a level bridle instead of directly to the vessel, about 6 in. of total operating range is lost because the top-most and bottom-most gage connections are staggered to avoid interference with the bridle process connections.

Since gage glasses are used instead of a magnetic level gage, another 6 in. of total operating range is lost because of the difference in visible range of these technologies. Also, the level transmitter is installed so that its connections to the bridle are staggered with those of the gage glasses, meaning another 6 in. of operating range is lost.

Lastly, consider that this vessel has a high level alarm at 90% of the transmitter span and a low level alarm at 10% of the transmitter span. The transmitter span is 8.5 ft, so this equates to another 20 in. of operating range that is lost since the level should not exceed the alarm limits during normal operation.

While this vessel has a height of 12 ft, only the middle 6 ft and 10 in. of the vessel represents usable volume (about 2,600 gal of the total 5,000-gal volume) within the alarm limits of the transmitter. The operating range has been significantly restricted by the types of level instruments used and the installation methods.

Incidentally, doing a quick calculation of the 2,600 gal of usable volume between the alarm points with 500 gal/min of throughput, the residence time of the usable volume is slightly over 5 min. So this is a case where vessel sizing Methods 1 and 2 would both have yielded essentially the same result. By using Method 2 to check final sizing, this confirms that the initial sizing with Method 1 was appropriate and does not need to be revised.

Compare this result to the same vessel where best practices for level instruments have been applied.

Best practice. Figure 6 depicts the same vertical vessel, but equipped this time with a single, magnetic level gage and a level transmitter, both with their own connections to the vessel rather than using a level bridle.

Again, the process connections for the level instruments are approximately 12 in. away from the tangent lines, so the nozzles would be about 10 ft apart.

Because a magnetic level gage is used and is directly connected to the vessel, the visible range covers the entire 10 ft between the process connections. The level transmitter is installed so its span is the same as that of the level gage, so it can also measure the entire 10-ft span. The alarm limits at 90% and 10% of the transmitter span will result in a loss of 24 in. of operating range.

In this case, the vessel has a height of 12 ft and the middle 8 ft of the vessel represents usable volume (about 3,000 gal of the total 5,000-gal volume) within the alarm limits of the transmitter. The operating range and usable volume have been increased by 15% over the previous case just by changing the types of level instruments used and how they are installed.

Although this vessel is the same size as that in the previous example, this additional 15% usable volume can allow for greater tolerance to a process upset without resulting in an alarm or a shutdown condition simply by allowing the operator and the control system to view and utilize a greater portion of the vessel.

Also important, the total installed cost of this system is expected to be less than that in the previous example, because it requires no bridle piping, fewer valves, fewer total welds (but more shop welds are required for the additional vessel nozzles) and fewer instruments. It also takes up less physical space that might have otherwise increased the access platform size.

Final notes

It is important to consider how a vessel will be instrumented in order to ensure the vessel size is appropriate. Considerations in evaluating the vessel and level instruments holistically should include the following:

- The process intent of the vessel
- Cost-effective vessel design
- Level instrument technology selected and installation method
- Time required for the operator to respond to alarms and regain control before safe operating limits are reached
- Physical access and platforms required to view and maintain the instruments, the block valves, and the vent/drain valves
- Coordination of the level bridle design (if used — not normally recommended) with the instrument installation and physical access requirements

These considerations will often go beyond the information available to just the process and mechanical engineers, and will need to include process control engineers, operators, and piping designers to provide the most safe, reliable, and cost-effective system possible. ■

Edited by Dorothy Lozowski

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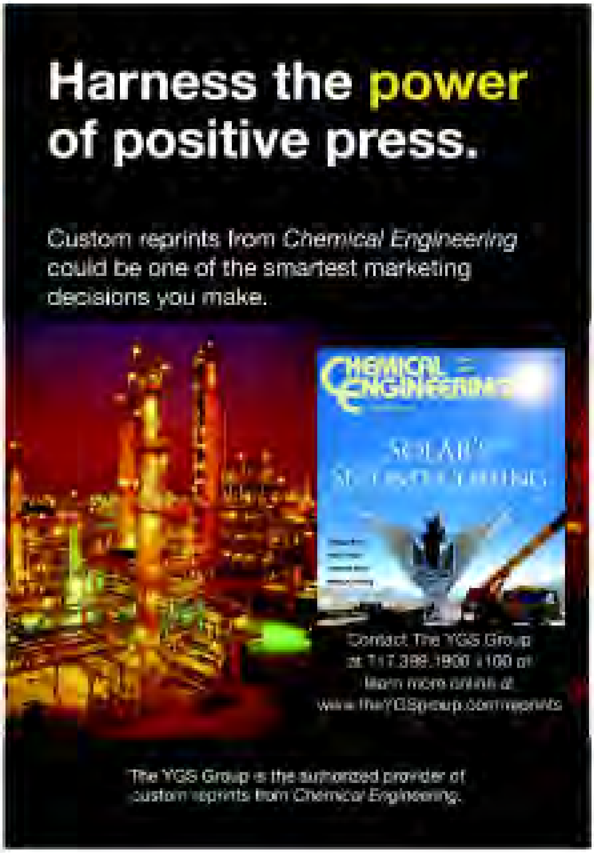

Choosing Oil-injected Screw Compressors

Follow this guidance to improve the design, performance and reliability of these workhorse machines

FIGURE 1. This medium-sized screw compressor is driven by an electric motor and installed on a steel frame skid. The top suction and side discharge nozzle arrangement is quite common in mid-size and large machines (typically >0.8 MW). Thermal and pulsation considerations are important when designing and installing the screw compressor's piping arrangement, particularly with regard to the discharge line design. Oil system and auxiliaries are typically included on the same skid

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Screw compressors are widely used throughout the chemical process industries (CPI) and represent a substantial percentage of all positive-displacement compressors that are now sold and in operation in CPI facilities. In general, screw compressors are efficient, reliable, simple and compact compressors that are competitive from both an initial cost and an operating cost standpoint compared to other types of positive-displacement compressors over a range of operating conditions.

Screw compressors have many inherent advantages over other compressor types, especially in the low- to medium-flow range. These positive-displacement compressors draw a constant inlet volume, meet varying differential pressure requirements, and are not significantly affected by changes in gas conditions, such as changes in density or gas composition.

And while screw compressors may transmit relatively low-level pulsations, they do not generate the types of severe pulsations that are typical of reciprocating compressors. Also, cylinder valves and valve unloaders — which can be problematic — are not required.

Two design options

Two types of twin screw compressors are available:

- Oil-free (dry type), and
- Oil-injected (wet type)

Although both types use screw rotors, they are significantly different, and this article focuses on oil-injected screw compressors.

Oil-injected (wet type) screw compressors. These are the newest and most advanced types of screw compressors. Compared to their dry alternatives, oil-injected screw compressors enable much higher compression ratios, simplified mechanical design (mainly because they do not require timing gears), more-efficient operation and advanced reliability.

Oil-free (dry type) screw compressors. In oil-free (dry-type) screw compressors, the gas is compressed entirely through the action of the screws, without the assistance of the oil. Timing gears ensure that the male and female screw rotors maintain precise alignment. These compressors usually have lower maximum-discharge-pressure capability, and they need relatively high rotational speeds. In many cases, they require gear systems to increase the driver speed. Because they are more complex to manufacture and cost more than oil-injected screw compressors, dry-running compressors tend to only be used where entrained oil carry-over (even in very small quantities) is not acceptable.

The benefits of oil injection

Today, oil-injected screw compressors have largely replaced reciprocating compressors and centrifugal compressors for many CPI applications. The general arrangement for a small, oil-flooded screw compressor package (Figures 1 and 2) requires a compressor and motor installed on a horizontal oil separator. Medium and large machines (those above 0.8 MW) are typically installed on skids.

In an oil-flooded screw compressor, lubricating oil is deliberately injected into the gas-compression space and is allowed to mix freely with the process gas. The oil acts as the coolant, enabling the gas-discharge temperature to be controlled. During operation, differential expansion between the screws and the casing — or more precisely, the contacting surfaces that are separated by the oil film — that results from thermal growth can reduce the running clearances and create interferences between the casing and the screw rotors. By injecting a calculated flow of oil, the discharge temperature can be reliably controlled (usually in the range of 75–110°C), and the oil film can help to minimize differential expansion between the contact surfaces. This permits relatively large pressure rises across each stage of the compressor and obviates the need for jacket or rotor cooling.

For optimum efficiency, internal clearances within the compressor must be kept as small as possible. The injected oil also serves as a sealant, partially filling the clearance between the screw rotors and casing.

The injected oil also acts as a lubricant enabling the main rotor (the drive rotor, which is generally the male rotor) to drive the female rotor through a film of oil. With this design, oil-injected screw compressors do not require a timing gear between the rotors to ensure that there is no metal-to-metal contact. This results in a simpler and less expensive compressor design, compared to dry screw compressors that require a timing gear.

This lubricant effect also permits the use of a slide valve, which acts as the capacity-control system. The valve is capable of controlling volume flow between typically 20–100%, providing one of the most flexible compression systems available.

The lubricating oil also acts as a corrosion inhibitor, protecting the compressor from the constituents in the compressed gas. This feature enables oil-injected compressors made from conventional materials (such as carbon steel or grey cast iron) to be used for difficult gases (those containing sulfides, chlorides and so on).

The oil acts as a noise dampener. Combined with the fact that screw compressors have relatively low rotor-lobe passing frequencies, this enables most installations to meet permitted noise levels without extensive sound attenuation.

Additional design aspects

Generally, grey cast iron is suitable for the compressor casing, for operating pressures to 15 barg. Casings made from spheroidal graphite (SG) iron can be used for pressures up to around 35 barg. Proper steel grades should be used for pressures above 35 barg.

Figure 3 shows a vertically split, oil-injected screw compressor in the workshop for repair. In the start of the suction stroke, gas enters the rotor flutes. The suction port is sealed from the gas-filled flutes by the meshing of the next lobe. The trapped gas is then briefly moved axially along, until the flutes reach the end wall and the compression stroke commences.

The volume trapped in the flutes is reduced until trapped gas reaches the discharge port. The volume ratio is an inherent physical feature of a given screw compressor design. The discharge pressure of screw compressors is limited by various factors, including the gas discharge temperature, the rotor length-to-dia. ratio (*L*/*D* ratio), the material and casing design (the common limit is below 70 barg).

FIGURE 2. In this medium-sized screw compressor, the effects of injected oil result in relatively lower speed (compared to comparably sized, dry-type screw compressors), which enables direct connection between the electric-motor driver and the screw compressor. This eliminates speed-increasing gears, which are usually required in dry-type screw compressors

Rotor speeds are such that dynamic balancing is required for proper vibration control. Screw rotors usually rotate around 10–25% below their critical (resonance) speeds.

Rotors made from forged carbon steel are commonly used for all non-corrosive applications. High-alloy steels (such as 13% Cr and 4% Ni) tend to be used only for corrosive or other special applications. Nearly all stainless steels are prone to galling. Particular attention is required for screw compressors that have relatively low operating clearances (around or below 80% of commonly used clearances) when using stainless-steel or alloy-steel materials. Alloy steels and stainless steels are also rarely used because of high price, operational problems and lack of proper references. Rather, for most screw compressors, a suitable carbon-steel grade is typically specified.

Screw profile options

Various screw profiles are available. Selection depends on the process conditions, and the anticipated flowrate and pressure conditions. The most common profile is called the asymmetric 4+6 screw (which has 4 male lobes and 6 female flutes). The screw configuration with a 4+6 asymmetric profile that is designated *‘A’* is predominantly used for relatively large oil-injected screw compressors (such as those above 0.3 MW). The screw configuration with a 4+6 asymmetric profile that is designated *‘D’* is predominantly used for small and medium oil-injected screw compressors (such as those around and below 0.5 MW). Figure 4 shows the rotors of a screw compressor.

Some vendors provide screw rotors with a range of coatings to improve wear resistance and maintain the desired seal clearance. But, there are diverse opinions on the value of such coatings because of costs, operational problems, relatively low life and some reported reliability issues.

In general, rotor coating is not generally recommended — except for the purpose of renewing clearances during machine repair or screw rotor rehabilitation. Because the metal surfaces in modern screw-type compressors are configured in a three-dimensional spiral slope, machining them to precise dimensions during repair is very difficult. Thus, some modern polymeric coatings that match rotor clearances have been used successfully in recent years for screw rotor repair. Compared to other repair or replacement options, rotor coating can be carried out at relatively low cost and low risk.

In a recent case study, the application of a screw rotor coating using a special polymeric coating on the contacting surfaces during machine renovation provided 4% improvement in machine efficiency.

In general, differential pressure on any stage should not exceed the maximum allowable values, depending on rotor design. As a rough indication, this limit could vary from as much as

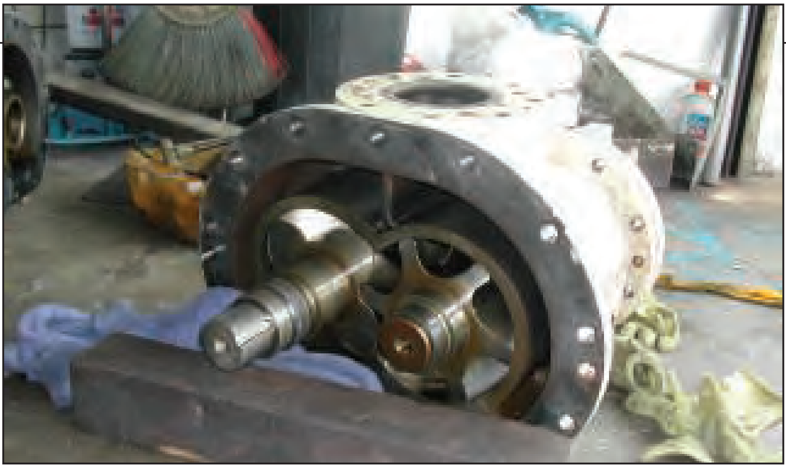

FIGURE 3. The male and female screw rotors, and internal and external casing are shown in this vertically split, oil-injected screw compressor. This machine uses an asymmetric 4+6 screw rotor arrangement (4 male lobes and 6 female flutes), the most common screw profile in various applications. The suction nozzle is located at the top

35 bar for a ratio of length-to-diameter (*L*/*D*) of 1, to as low as 7 bar for *L*/*D* = 2.3 for a screw configuration with a 4+6 profile. The reason for this limitation is rotor deflection. The larger the *L*/*D* value, the greater the bearing span and thus the larger the rotor deflection. The use of rotors with higher rigidity (those with, for instance, a 6+8 profile combination and *L*/*D* = 1), can provide a differential pressure capability of as much as 50 or 60 bar.

The male and female rotors of a screw compressor theoretically contact each other along a sealing line. There is a clearance distribution along this sealing line, which influences the efficiency and reliability of the screw compressor. The amount of clearance depends on a variety of factors, including profile error, thermal deformation, center displacement and the relative rotational angle between rotors.

Medium and large-size screw compressors (roughly above 0.7 MW) typically incorporate sleeve bearings (also called journal bearings or sliding bearings) and self-adjusting, multi-segment thrust bearings. For difficult services, specially designed bearings may be used.

Since the highest loads in a screw compressor are axial, the thrust bearing is the component that is most susceptible to premature failure. The compressor should therefore be protected by using embedded temperature sensors mounted in the thrust bearings (the axial bearings). The design should allow bearings to be easily removed without requiring the removal of the screw rotors or the upper half on the horizontally split machine. Oil-injected screw compressors require only one seal, where the driver shaft enters the compressor casing.

Gas contamination (that is expected during various modes of operation) should be accurately defined by the user. For example, handling sour gas or other corrosive agents will dictate material types, oil type, and other key machine or operating considerations.

The ability to achieve the required outlet pressure, regardless of the gas molecular weight, gas composition or operation changes, is another key criteria for CPI applications. For instance, a screw compressor that starts on nitrogen and then gradually brings in a hydrogen-rich gas mixture should not change in performance. This operational flexibility is a great advantage compared to dynamic compressors, such as centrifugal compressors.

The screw compressor module must be fully integrated into the CPI plant system. In other words, in the screw compressor package — particularly for the inlet system and discharge facilities — the oil type and some other auxiliaries should be customized to meet the site-specific needs of each process application.

Meanwhile, if particulate contamination is present in the gas, solid or liquid, especially in large quantities, erosion and lubrication problems may result. Thus, a properly sized knock-out drum, separator and filter should be installed upstream of the screw compressor inlet.

Managing lubricant oils

Oil-injected screw compressors are typically fitted with a slide valve, which permits the suction volume to be reduced. This system gives operators step-less control of capacity, while maintaining high overall efficiency.

Oil-injected screw compressors are usually constant-speed machines. The use of a throttle valve to control suction capacity may be used only for small screw compressors, since this approach can reduce efficiency and also may cause some operational and reliability problems, such as excessive discharge temperature, internal leakage and excessive rotor deflection.

The maximum temperature that gases are allowed to reach is usually determined by the lubricant type (*CE*, January 2012, pp. 44–48). For mineral lubricants, the normal maximum temperature maximum is in the range 75–90°C (sometimes up to 95°C). Some synthetic lubricants will permit this discharge temperature to be extended up to 120°C.

The quantity of oil injected into the compressor is adjusted to make sure that the combined gas and lubricant discharge temperature does not exceed the specified values. Mineral oil viscosity drops rapidly with rising temperature. Also mineral oils can be affected by some gases (such as oxygen), especially above 80°C. In general, compared to mineral oils, synthetic lubricants can operate at higher temperatures because they have high viscosity indices; thus, they can be used (and are more stable) over a large temperature range. Today, synthetics are being increasingly used in screw compressors.

All oil-injected screw compressors require an oil-separation system, which is usually combined with the lubrication system. Depending on the selected lubricant, oil pumps, cooler and filters, the system must be designed with an appropriate oil separator retention time and proper oil-temperature control loop. The oil separator vessel is generally equipped with suitable internals and wire mesh demister devices (or alternative devices) to separate the oil that is entrained in the gas stream.

The lower part of the discharge vessel (oil-separation vessel) serves as the reservoir for the oil management system and can usually be designed

FIGURE 4. This photo shows an example of the screw rotors in a screw compressor. Again, this image shows a 4+6 screw rotor arrangement

with an oil retention time of 60 to 120 seconds. When the screw compressor is being used to compress particularly corrosive gases, it is common to line the upper part of the vessel (above the demister pack) with an epoxy or glass-based material.

The discharge pressure of the oil pump is selected based on compressor discharge pressure (adding a margin, usually 2–4 bar), to ensure adequate oil injection.

Use in CPI settings

Today, medium-size screw compressors (say, in the 0.3–1.5 MW range) are widely used for fuel desulfurization processes. Although the main constituent of the gas compressed in this service is hydrogen, small amounts of corrosive sulfur hydride are usually present, along with relatively heavy hydrocarbons, such as propane and butane.

Relatively heavy hydrocarbons can dissolve into the lubricant and reduce its viscosity. This can cause breakage of oil film at the bearings, which can increase the wear rate, even leading to premature failure. Today's synthetic oils dissolve only small amounts of relatively heavy hydrocarbon gas, thereby allowing screw compressors to be successfully used in fuel desulfurization and other CPI applications.

The compressor has to accommodate a variety of operating conditions and various gases with different properties, including the nitrogen that is used during startup. Traditionally, reciprocating compressors have been used for fuel-desulfurization applications. However, the use of oil-flooded screw compressors is on the rise in this area because of their high reliabilities and more compact and less expensive packaging.

For most applications, a spare is not required for screw compressors. By comparison, because reciprocating compressors have relatively low reliability, operators often keep one in use and one ready in standby mode.

There have been some reports and problems related to the use of oil-flooded screw compressors in flare gas recovery (FGR) applications at various CPI facilities. Such problems primarily result from the dynamic composition of the flare gas, which increases the challenge to select an ideal lubricant — one that can operate successfully in the face of this wide range of gas compositions. As a result, for FGR applications, it is important to use a robust and reliable forced-feed lubrication system with carefully designed loops to fully control lubricant temperature and other key parameters during operation.

The use of screw compressors is also on the rise for systems handling refinery vacuum-offgas, which usually contains corrosive sulfur hydride. Such service requires continuous operation, and if the compressor goes down, the offgas normally has to be flared (or the plant shut down). Over time, the lubricant will become contaminated with hydrogen sulfide, some hydrocarbons, water and other substances.

If the screw compressor is fitted with ordinary (standard) bearings that are made from conventional materials, there is also an increased risk of catastrophic failure (due to corrosion-related problems). High levels of hydrogen sulfide, combined with water condensation, can result in surface spalling and stress cracking of bearing raceways. Standstill periods can exacerbate the problem.

The most severe damage usually occurs in angular contact ball bearings, which experience the heaviest loads. In a recent case study for a refinery screw compressor handling gas with hydrogen sulfide, bearing balls actually split in half after several months of operation.

When using conventional bearings, the screw compressor's mean time between failures in sour service could be less than 4,000 hours (less than six months). However, by choosing advanced bearings technologies, such as bearings made from super-rugged, high-nitrogen, martensitic stainless steel and featuring silicon nitride ceramic rolling elements, the installment was able to achieve continuous trouble-free operation beyond 30,000 hours (3–4 years). Generally, advanced bearings should be considered for screw compressors in all applications that are likely to handle aggressive gases.

One alternative is the use of dual-circuit oil injection, which involves the use of separate oil circuits for the compressor oil injection (the working chamber) and the bearings. Not many vendors offer these since they tend to be a complex, less well known, and more-expensive solution. Nonetheless, where appropriate, dual-circuit oil systems should be considered during the specification or material-requisition stage of the bidding process.

A tandem-screw-compressor includes two screw compressor stages. Such a system may be treated as a single compressor, because the drawn gas from the first stage is fed directly into the second stage without being discharged to the outside of the compressor. An intercooler is generally not required. Thus, only one set of components — such as a motor (driver) and oil separator — is required to achieve the two-stage compression, enabling the two-stage compressor to be treated the same as the single compressor. Tandem-type compressors are very compact and efficient machines. They tend to be used in relatively high-pressure (around 40–70 bar) CPI applications.

As discussed above, special modifications and considerations may be required to enable the use of oil-injected screws in specific types of CPI gas services (such as sour gas service). In general, standard discharge oil separators that are used downstream of the screw compressor (providing standard 60 to 120 seconds of retention time) do not allow specific gas components (such as sour gas components) to be sufficiently vented from the oil reservoir. Over time, this can lead to oil contamination, screw component damage and more-frequent oil changes.

For special applications, suitable liquids other than oil (for instance, water, some acids, and so on) may be injected into the compressor. For instance, modern, water-injected, two-stage screw compressors are popular for coke gas units. Other types of compressors (such as centrifugal compressors) are not successful in coke gas processing service because they enable rapid polymerization.

Reliability and machine data

When using screw compressors, the keys to reliability are modern seal and bearing designs, the use of the most appropriate lubricants and proper modularization. Similarly, the use of condition monitoring should focus on the following operating parameters, as they provide a good indication of the operating condition of the system:

- The screw rotor (particularly the flow trend and efficiency trend)
- The radial and thrust bearing (mainly the bearing temperature and casing vibration)
- The seal and oil systems

Reduced gas flow can indicate a problem, such as slip increase (an increase of internal leakage due to enlarged clearances or damage). During monitoring of flow, the rotor speed, suction throttle valve (if used), slide valve and bypass position should be kept constant particularly when evaluating flow trending. Off-design operation should be avoided at all times. High-temperature operation (above the rated temperature) may lead to damage of the screw rotor and permanent efficiency loss.

When specifying screw compressors, the manufacturer should be given the freedom to optimize the machine configuration. The requisition data sheet should show which parameters are mandatory and which are desirable. Limiting values should be given as necessary. The data sheet should include all identifiable process duties. One duty should be designated Rated Point; this defines the extremes that are necessary to meet all specified operating points (including minimum and maximum points). It dictates the size of the compressor train. This rated point is typically specified by identifying the normal operating point and adding margins curves of all duties. ■

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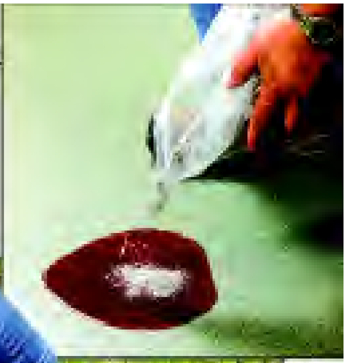
Spill Response


Brady


Ultra-Tech International


New Pig

This filter captures hydrocarbon spills while passing rainwater

Ultra-Filter Pads (photo) are suitable for use on construction sites, oil fields and other areas where spill containment is needed but shelters or structures are not available to keep out rainwater. The filters capture oil, grease and other hydrocarbons while allowing clean rainwater to flow through. The filter's polyethylene and polyvinyl construction is lightweight but durable, says the manufacturer, and it folds for quick and easy storage or transport. Replaceable Ultra-X-Tex liners quickly capture oil, grease and fuel leaks and spills. Two-inch foam sidewalls provide structure and help contain storm water during periods of quick or large amounts of rainfall, ensuring the liner has sufficient filtering time. — *Ultra Tech International, Inc., Jacksonville, Fla.*

www.spillcontainment.com

Divert and collect spills before they become a problem

The Pig Low-Profile Leak Diverter (photo) was recently introduced for catching leaks in tight spaces and channeling them away before they damage the environment or equipment, or create a slip hazard. The Low-Profile Leak Diverter is made of a durable, vinyl-coated, non-permeable material that resists punctures, mildew and degradation. The diverter's compact size allows workers to target spills — even those in tight spaces — and can be used as a long-term maintenance solution, or as an emergency response when a leak is discovered. The diverter is resistant to ultraviolet radiation for up to 12 months, making it suitable for outdoor use, and is flame resistant for added safety. The unit accepts a standard 3/4-in. hose connection to safely channel liquid to floor drains or to a collection container. — *New Pig Corp., Tipton, Pa.*

www.newpig.com

This high-absorbancy sorbent is made from recycled material

This company has expanded its SPC absorbent product line to include the new Re-Form Granular absorbents. This absorbent is made from 100% recycled post-consumer paper waste, and absorbs — on contact — oils, solvents, lubricants, coolants, water and most non-aggressive liquids. With a one-to-one absorbancy ratio and low moisture content, Re-Form Granular absorbents can absorb their own weight in oil and can fully retain spills. They were tested to have one of the highest bulk densities among alternative granular sorbents, thus they do not blow away in the wind or other adverse weather conditions. The granular absorbent has no detectable level of crystalline silicate or other hazardous materials. They are pH neutral, which decreases the probability of reacting with volatile liquids, and are virtually dust-free, leaving no stains or visible residue. They are also low in ash content, which makes them easier to incinerate and reduces the amount of residue sent to landfills. — *Brady Corp., Milwaukee, Wisc.*

www.bradycorp.com ■

Gerald Ondrey

Note: For more information, circle the 3-digit number on p. 50, or use the website designation.

Control Strategies Based On Realtime Particle Size Analysis

Practical experience illustrates how to achieve better process control

Jeff DeNigris
Malvern Instruments

Alberto Ferrari
Ferrari Granulati

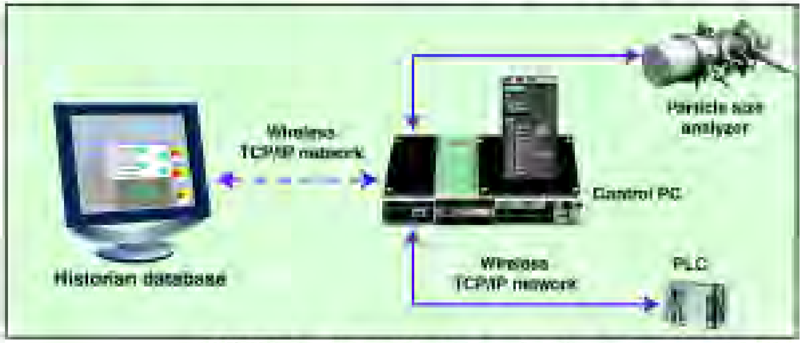


FIGURE 1. Integrated system architecture is illustrated here for an automated mill with an online particle-size analyzer

The pursuit of manufacturing excellence is a strong theme across all of the chemical process industries (CPI). Improving operations by eliminating variability and waste, a cornerstone of the six sigma approach, is essential for competitive performance in the global marketplace. In addition, of course, health, safety and the environment (HSE) remains the subject of intense scrutiny and concern. Within this climate, automation of both analysis and control, can be highly advantageous.

Particle size analysis is a measurement technique that has already successfully completed the transition from laboratory to line and become an established part of the automation toolkit. Reliable, online particle-size analysis systems are now commercially proven across a number of sectors, on both wet and dry process streams, with all aspects of project implementation well-scoped and understood. With this maturation has come greater accessibility to sectors that previously faced technical or financial barriers to adoption. Requirements are different in each and every case, from a simple single PID (proportional integral derivative) control loop to multivariate statistical control or the rigors of operation within a highly regulated environment. Nevertheless, the building blocks needed to fashion an optimal solution are there, for the vast majority of applications.

This article draws on experience from different industries as they apply realtime particle-sizing technology, to demonstrate the various control strategies that it supports and the benefits that can be derived.

Note: With an online system the analyzer is typically installed on a dedicated loop fed from the process line, while with an inline instrument the analyzer sits directly in the bulk process flow. Both approaches provide continuous realtime data so for the purposes of this article the term online has been used to cover both types of installation.

Moving toward realtime

It should be stated from the outset that offline analytical capability remains vital. Essential during research, it is also frequently the norm for final quality control checks. Furthermore, certain techniques that yield critical information have yet to move successfully to the process environment. That said, for routine process monitoring an offline regime is far from ideal and online measurement, if available may be the preferred option.

Laser-diffraction particle sizing exemplifies a number of commercially proven, online analytical technologies with established credentials for realtime monitoring. Today it is possible to tailor an online particle-sizing solution that closely matches user requirements, from turnkey integration within a sophisticated control platform to sensor-only purchase for the inhouse implementation of simple closed-loop control. This ready availability of realtime measurement presents an important opportunity to realize a number of important practical gains, even before considering the issue of process control.

Full automation — from sample extraction through the delivery of results to a control system — means that online analysis completely eliminates the issue of operator-to-operator variability. In addition, the ability to analyze a much higher proportion of the process stream improves the statistical relevance of the data. Equally important is that the benefits of automation come with a dramatic decrease in the amount of manual labor required for analysis, and a significant reduction in the containment and exposure risks associated with manual sampling and analysis.

Investing in realtime measurement technology may be justifiable simply on the basis of these practical benefits, especially where the envisioned set-up is relatively simple. The cost of installation is offset by savings in manpower, and the operational team maintains manual control exactly as before, using the online system solely for particle size monitoring. While this approach may deliver improved operations and variable cost gains, it fails

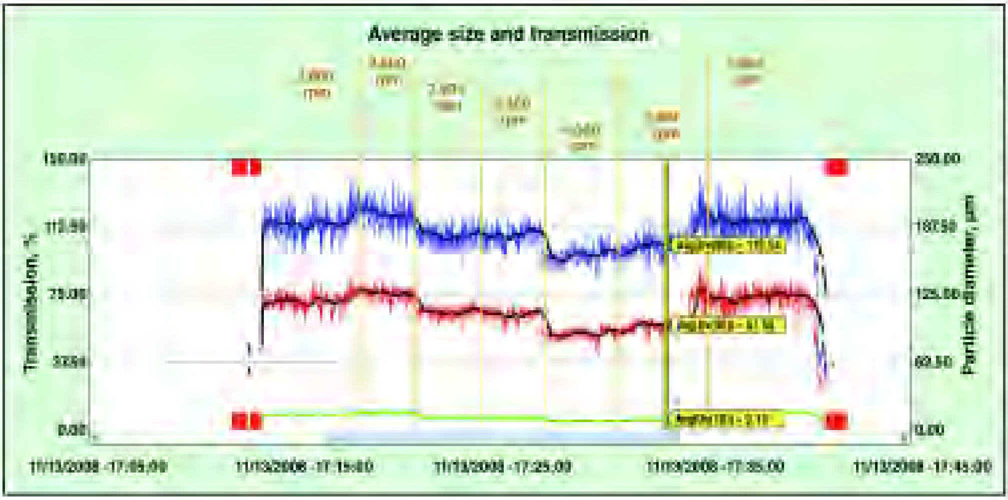


FIGURE 2. For simple, closed-loop control, online analysis can be used to track particle size changes that are induced by varying mill rotor speed over time

to fully realize the potential that online instrumentation offers in opening up the route to automated control. An efficient control-automation strategy fully exploits the information stream provided by realtime measurement and maximizes return on investment.

Simple closed-loop control

The simplest option when implementing automated control on the basis of continuous data from an online instrument is usually a single variable PID control loop. Such an approach can prove highly productive and be an efficient way of automating existing manual control strategies. Even the implementation of one automated loop changes the process from being fixed to becoming responsive. A fixed process translates, or magnifies, upstream variations on to the product or downstream process; a responsive one either erases or reduces their impact.

For milling, a common approach is to automatically vary either mill speed or downstream separator variables (classifier speed, for example) to meet the product specification (as in the example below). An exactly analogous strategy in emulsification processes allows key variables, such as pressure, to be automatically manipulated in order to control droplet size. Since online particle-size analyzers are commercially available for both wet and dry process streams, any unit involving comminution to a defined particle size can potentially benefit from this very basic type of automation.

Case study: Simple closed loop control on the basis of realtime data. An automated mill system recently installed at a commercial pharmaceutical manufacturing site is shown in Figure 1. It was developed as a widely applicable, validated alternative to manual mill control using offline particle-size measurement. One routine operation in the company is milling an active pharmaceutical ingredient (API), typically recovered through crystallization, to a defined particle size. The particle size distribution of an API is often a critical quality attribute because of its impact on clinical efficacy and drug product manufacturability.

The comminution mill has fast dynamics, making rapid and continuous data acquisition and interpretation essential. Since the selected online particle-size analyzer has a measurement rate of four complete particle-size distributions per second, it can efficiently track even this swiftly changing process in fine detail.

This fully integrated system uses upgraded programmable-logic-controller (PLC) code and proprietary software to handle data exchange between the main hardware units. The operator interacts with the central controlling PC via the mill HMI (human machine interface) and can do the following: input set points; start and stop the mill or analyzer remotely; perform background tests; and receive particle size results. A closed control loop links particle size with mill rotor speed, the principal operating parameter for size manipulation (Figure 2). A 30-s rolling average Dv50 (median particle size) figure from the online particle-size analyzer drives this loop.

To test the response of the system, the setpoint for the loop was reduced from an initial 58 microns to 50 microns, and then back up to the original value. Despite the absence of comprehensive loop tuning — proportional (P) only control was used at this stage — the results were good. Steady operation at 50 microns was established just 30 s after the change was made, and the final transition was complete in under 2 min.

To mill a new batch with this system, the operator simply selects the target particle size and feeds material into the mill. Control is then sufficiently tight to largely eliminate the production of out-of-specification material. Contrasting this control scheme with offline or manual analysis quickly brings the multiple benefits into sharp focus:

- When using offline particle-size analysis, each new batch required a potentially lengthy iterative process to determine the appropriate rotor speed, in order to meet the defined specification. Eliminating this step has saved both time and material.
- Prior to automation, the rotor speed was fixed for each batch, based on a test sample. However, if the sample was not representative of the batch, or if segregation had occurred, the rotor speed would be less than optimal and the product

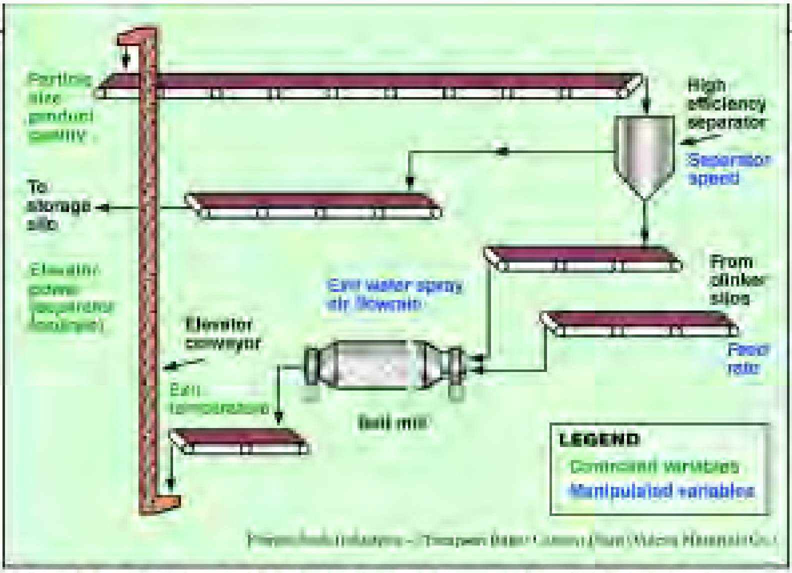


FIGURE 3. This upgrade to a cement finishing circuit for a milling clinker (and gypsum and limestone) helps achieve the fineness required to meet final cement specifications

inconsistent. Since the automated mill responds to, and compensates for, any variability in the feed stock, the product particle size has become extremely consistent.

Multiple control loops

The next step beyond a single control loop involves multiple, simple control loops, which enable the parallel manipulation of a number of variables to simultaneously meet product quality, variable cost and throughput goals. For example, at its plant close to Verona in Italy, Ferrari Granulati mills very fine marble powders of exemplary quality [1]. Three discrete products are marketed with Dv50s in the range of three to eight microns. Here, online particle-size analysis has been used extensively to develop the design of the mill and a control strategy for the milling circuit (mill and associated classifier).

The adopted strategy relies largely on fixing a number of process variables, at values defined through detailed optimization trials that are based on realtime measurement. These values have been defined for each product. Two independent, automated control loops are, however, applied to optimize mill performance and maximize plant throughput on an ongoing basis. One loop maintains a prescribed powder depth on the table of the vertical roller mill to ensure efficient comminution and prevent excessive wear of the mill. The other controls the rate of fresh feed to the mill with reference to the recycle rate from the classifier, to ensure that the total feedrate to the mill remains constant. In combination with the fixed operating strategy these loops ensure that exceptional product quality is achieved at competitive cost.

As with the previous example, the architecture of the control loops employed here is relatively simple: one single process variable manipulated on the basis of a single measured variable. Nevertheless, they are highly effective with each loop efficiently targeting one specific aspect of process performance.

Multivariate process control

Just as a seasoned operator bases manual control decisions on every piece of relevant information, the most sophisticated automated control relies on an array of data, rather than a single input. Multivariate process control systems take in and use data from a number of sources and, in combination with a process model, provide multiple outputs, simultaneously manipulating various operational parameters. Such systems work within well-defined boundaries to target optimal operation at all times.

Quite recently, steps have been taken to reduce one of the barriers to implementing multivariate process control: the difficulty of integrating analyzers from different suppliers. The new OPC Foundation Analyzer Device Integration (ADI) specification [2] provides a common standard for instrument manufacturers. Into the future this should ease the integration of both process and laboratory systems. Enabling software, based on this specification, is already available commercially. These advances will reduce the difficulty and cost of implementing customized multivariate control strategies, bringing the potential rewards within the reach of more manufacturers.

Case study: Multivariate control of a heavy commodity milling circuit. In 2006, Vulcan Materials Company (Birmingham, Ala.) made the decision to transform control of its cement finishing circuit (Figure 3). The project involved the three following significant changes:

- Switching from Blaine measurement to laser-diffraction particle-size analysis, with the intention of more precisely targeting cement performance and accessing online technology
- Adopting online, rather than offline analysis for process control
- Selecting and installing a powerful model-predictive-control package to automate process control

Vulcan Materials installed a proprietary solution for multivariate control and an online laser-diffraction particle-size analyzer. At the heart of the control package is a multivariate process model that is tuned using plant data to accurately predict plant performance from a range of inputs. Automatic manipulation of process variables, on the basis of these predictions, achieves plant performance targets, which are as follows:

- Maintain product quality
- Reduce variability and improve operational stability
- Maximize fresh feedrate subject to equipment constraints

The process model runs in real time, employing an integrated steady-state optimizer and dynamic controller to drive the system toward optimal operation within the above constraints. The impact of changing manipulated variables is projected into the future; predictive control ensures that multiple performance targets are met simultaneously and that process outputs are as close as possible to desired reference trajectories. Optimization procedures are repeated each time process values are re-read, following a change, in order to maintain the future prediction-horizon period. This is termed "realtime receding horizon control" and is characteristic of model predictive control.

Here, a number of loops are operating in tandem (Figure 3). Manipulating clinker feedrate and separator speed controls product quality. These same two parameters are also used, together with air flow through the mill, to drive separator feedrate (measured as elevator power) toward a defined high limit. Air flowrate through the mill and exit water spray variables control the temperature of exiting material. Finally, a stabilizing loop minimizes a function defined as "mill condition", calculated from the rate of change of elevator and mill power. The online particle-size analyzer measures product quality in real time, providing vital information that is used by the model in combination with an array of other process measurements.

The installed solution has pushed the circuit into a new operating regime, uncovering better control strategies than had been identified through manual operation. Significant benefits have accrued, including the following:

- A 20% reduction in specific energy consumption
- An improvement of 15% in one-day strength levels (a primary performance indicator)
- An increase in throughput in excess of 15%

These savings are not individually attributable to either the analyzer or the control package but arise from symbiosis between the two, which has unlocked the full potential of each. The payback time for the entire project is estimated at just over one year, based on energy savings alone.

Conclusions

The automation of process analysis and its closer integration with plant operation presents an opportunity for improved control, reduced risk and financial gain. For online particle-size analysis proven automated systems have developed to the point of widespread availability, making this opportunity financially and technically feasible across a broad range of manufacturing sectors. Options for investment now range from fully integrated turnkey solutions to sensor-only purchase for in-house implementation.

Continuous, realtime analysis of a critical variable, one that directly influences product performance, provides a platform for developing and implementing the very best automated control strategies for a given application. Such strategies deliver multiple economic benefits in the form of reduced waste, increased throughput, enhanced product quality, reduced manual input and lower energy consumption. Automating control realizes the full potential of realtime analysis, extracting maximum return from an investment in process analytical technology. ■

Edited by Rebekkah Marshall

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Jeff DeNigris joined Malvern Instruments in 2005 as national sales manager, process systems to focus on supplying online, realtime particle size analyzers to the pharmaceutical, fine chemical, mineral, toner and cement markets (Address: ; Phone: XXX-XXX-XXXX; Email:). He graduated in 1989 with a B.S.M.E. degree from Widener University, College of Engineering in Pennsylvania. He has spent most of his career in the manufacturing sector with top original equipment manufacturers of capital equipment. Particular focus was given to the plastics industry in the sales and marketing of recycling and reclaim systems, and to the design and implementation of granulation and separation systems.

Alberto Ferrari is the production manager of Ferrari Granulati S.A.S., a leading producer of marble chips and powders, situated in Grezzana (Verona) Italy. He is a specialist in the fields of information technology and industrial automation, working for five years as R&D manager in Dellas S.p.A., a producer of diamond tools, before returning to the family company in 1995. He continues to work to improve the efficiency of milling and sieving plants for calcium carbonate minerals.

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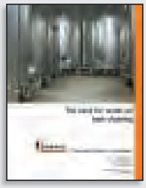

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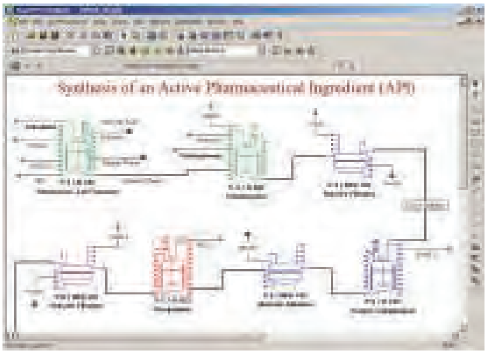

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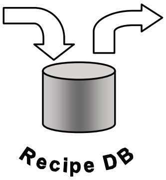

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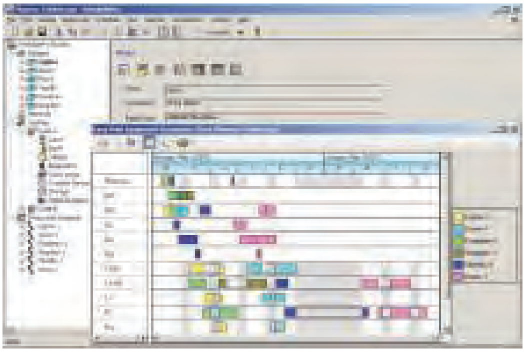

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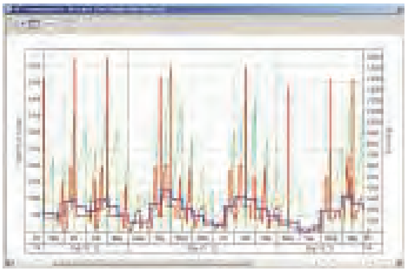

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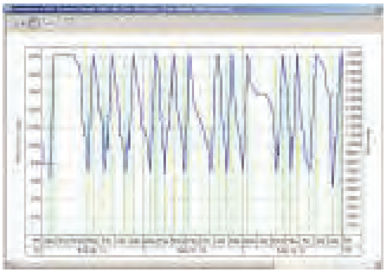

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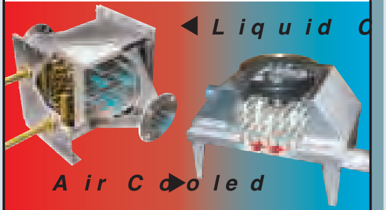

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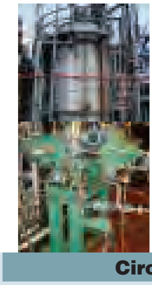

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58 Materials of Construction
59 Compressors

1	16	31	46	61	76	91	106	121	136	151	166	181	196	211	226	241	256	271	286	301	316	331	346	361	376	391	406	421	436	451	466	481	496	511	526	541	556	571	586
2	17	32	47	62	77	92	107	122	137	152	167	182	197	212	227	242	257	272	287	302	317	332	347	362	377	392	407	422	437	452	467	482	497	512	527	542	557	572	587
3	18	33	48	63	78	93	108	123	138	153	168	183	198	213	228	243	258	273	288	303	318	333	348	363	378	393	408	423	438	453	468	483	498	513	528	543	558	573	588
4	19	34	49	64	79	94	109	124	139	154	169	184	199	214	229	244	259	274	289	304	319	334	349	364	379	394	409	424	439	454	469	484	499	514	529	544	559	574	589
5	20	35	50	65	80	95	110	125	140	155	170	185	200	215	230	245	260	275	290	305	320	335	350	365	380	395	410	425	440	455	470	485	500	515	530	545	560	575	590
6	21	36	51	66	81	96	111	126	141	156	171	186	201	216	231	246	261	276	291	306	321	336	351	366	381	396	411	426	441	456	471	486	501	516	531	546	561	576	591
7	22	37	52	67	82	97	112	127	142	157	172	187	202	217	232	247	262	277	292	307	322	337	352	367	382	397	412	427	442	457	472	487	502	517	532	547	562	577	592
8	23	38	53	68	83	98	113	128	143	158	173	188	203	218	233	248	263	278	293	308	323	338	353	368	383	398	413	428	443	458	473	488	503	518	533	548	563	578	593
9	24	39	54	69	84	99	114	129	144	159	174	189	204	219	234	249	264	279	294	309	324	339	354	369	384	399	414	429	444	459	474	489	504	519	534	549	564	579	594
10	25	40	55	70	85	100	115	130	145	160	175	190	205	220	235	250	265	280	295	310	325	340	355	370	385	400	415	430	445	460	475	490	505	520	535	550	565	580	595
11	26	41	56	71	86	101	116	131	146	161	176	191	206	221	236	251	266	281	296	311	326	341	356	371	386	401	416	431	446	461	476	491	506	521	536	551	566	581	596
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13	28	43	58	73	88	103	118	133	148	163	178	193	208	223	238	253	268	283	298	313	328	343	358	373	388	403	418	433	448	463	478	493	508	523	538	553	568	583	598
14	29	44	59	74	89	104	119	134	149	164	179	194	209	224	239	254	269	284	299	314	329	344	359	374	389	404	419	434	449	464	479	494	509	524	539	554	569	584	599
15	30	45	60	75	90	105	120	135	150	165	180	195	210	225	240	255	270	285	300	315	330	345	360	375	390	405	420	435	450	465	480	495	510	525	540	555	570	585	600

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BUSINESS NEWS

PLANT WATCH

UOP's Oleflex technology selected by Dow Chemical

January 6, 2012 — UOP LLC (Des Plaines, Ill.; www.uop.com), a Honeywell company, has been selected by The Dow Chemical Co. (Midland, Mich.; www.dow.com) to provide technology to produce propylene at a Dow production site in Texas. Dow Texas Operations will use UOP's C3 Oleflex technology to produce 750,000 metric tons per year (m.t./yr) of polymer-grade propylene. The unit is scheduled to start up in 2012.

Jacobs secures contract with ISACC in Saudi Arabia

January 4, 2012 — Jacobs Engineering Group Inc. (Pasadena, Calif.; www.jacobs.com) has been awarded a contract by IDEA Soda Ash and Calcium Chloride Company (ISACC) to provide engineering and project management services for the construction of a $300-million soda ash (sodium carbonate) and calcium chloride production facility in Jubail's Second Industrial City, the Royal Kingdom of Saudi Arabia. Commercial operations are expected to start in the 1st Q of 2015. ISACC is setting up a new entity — Jubail Inorganic Chemicals Industries Co. — to run the new facility.

Topsøe signs contract for ammonia plant with Russian JSC Acron

December 22, 2011 — Haldor Topsøe A/S (Lyngby, Denmark; www.topsoe.com) has signed a contract with the Russian company JSC Acron for the design of a 2,060 m.t./d ammonia plant. The plant will be located in Veliky Novgorod, where an ammonia plant was under construction in the late 1980s. Due to the collapse of the Soviet Union, the construction of the plant was put on hold, but it will now be reconstructed. The new plant is expected to be in operation by the end of 2014.

Evonik to build a new methionine production complex in Singapore

December 16, 2011 — With the largest of its chemical investments to date, Evonik Industries AG (Essen, Germany; www.evonik.com) will invest roughly one-half billion euros in Singapore for the construction of a methionine complex with a capacity of 150,000 m.t./yr. The new plant is scheduled to begin operating in the 2nd half of 2014. The new construction and the expansion of four existing methionine plants will raise Evonik's total capacity for this amino acid to 580,000 m.t./yr beginning in 2014 — an increase of more than 60% in five years.

Air Products receives largest, single onsite ASU order ever for China

December 14, 2011 — Air Products (Lehigh Valley, Pa.; www.airproducts.com) has signed a longterm contract with Shaanxi Future Energy Chemical Co., and will build, own and operate an onsite air separation unit (ASU) in Yulin, Shaanxi Province, China. The unit will produce 12,000 ton/d of oxygen and significant tonnage volumes of nitrogen and compressed dry air for Shaanxi's coal chemical plant. The Air Products ASU trains are scheduled to be operational in 2014.

Koch Membrane to install wastewater system for steel plate manufacturer

December 12, 2011 — Koch Membrane Systems, Inc. (KMS; Wilmington, Mass.; www.kochmembrane.com) has been selected to supply membranes for a large-scale wastewater recycling system for Yingkou Medium Plate Co., in Yingkou City, Liaoning Province, China. The project will recycle the plant's wastewater to meet government discharge regulations and to conserve the region's fresh water supply. KMS will work in cooperation with Capital Engineering & Research Inc. (CERI), the engineering firm responsible for designing and building the system. The system is designed to handle an annual average flow of 80,000 m^3/d. Start-up is scheduled for early 2012.

Air Liquide plans new plant in North Dakota

December 12, 2011 — Air Liquide Industrial U.S. LP (Houston, Tex.; www.us.airliquide.com) has announced that it will build a new ASU to supply nitrogen to industrial users, particularly those in the oil and gas sector, in northwest North Dakota. The new ASU is slated to be operational by the end of 2012 and will be located in the Bakken reserves.

BASF investing $20 million in Vidalia, La. plant improvements

December 7, 2011 — BASF Corp. (Iselin, N.J.; www.basf.com) will be investing $20 million in its existing production facility in Vidalia, La. for activated alumina adsorbent products. This longterm improvement project will consolidate the company's regional manufacturing activities in a single, state-of-the-art operating environment. As part of this project, the company will increase its production capacity and operating capabilities in Vidalia, while transferring production from its smaller adsorbents plant in Port Allen, La., over time. As a result, the Port Allen facility is expected to discontinue operations in 2013.

MERGERS AND ACQUISITIONS

AkzoNobel to take 100 % control of Metlac Group

January 9, 2012 — AkzoNobel (Amsterdam, the Netherlands; www.akzonobel.com) plans to exercise the right to buy the remaining shares of Metlac, an Italian-based producer of packaging coatings. AkzoNobel currently is a shareholder in the Packaging Coatings Metlac Group. This position was inherited from the acquisition of ICI in 2008. The completion of the transaction is subject to antitrust approval. The company expects to finalize the acquisition in the 2nd Q 2012. Financial details were not disclosed.

DuPont and NexSteppe collaborate on feedstocks for bio-based products

January 6, 2012 — DuPont (Wilmington, Del.; www.dupont.com) and NexSteppe (Malibu, Calif.; www.nexsteppe.com) have entered into a collaboration to develop advanced feedstocks for biofuels and other bio-based products. The collaboration will focus on the development of new sweet sorghum and high-biomass sorghum hybrids that will create additional feedstock options for bio-based products. Under the agreement, DuPont has made an equity investment in NexSteppe, and will provide resources through its Pioneer Hi-Bred (Des Moines, Iowa; www.pioneer.com) business to help the company accelerate the breeding and commercialization of new hybrids of these crops in the U.S. and Brazil.

UOP creates dedicated natural gas and hydrogen business unit

January 4, 2012 — UOP LLC has established a new business unit dedicated to natural gas processing solutions and hydrogen. The new Natural Gas and Hydrogen business unit will increase UOP's focus on developing technology, equipment and adsorbents as well as the design and execution of projects for global natural-gas and hydrogen customers.

ABB invests in utility-scale concentrating-photovoltaic solar-power company

December 14, 2011 — ABB (Zurich, Switzerland; www.abb.com) has agreed to invest approximately $20 million as part of a $35 million financial round, for a substantial minority stake in California-based GreenVolts, a provider of turnkey concentrating-photovoltaic (CPV) systems. This technology complements ABB's recent acquisition of a stake in Novatec Solar, a leading provider of Linear Fresnel concentrating solar-power technology. ■

Dorothy Lozowski

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

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Economic Indicators

2009 🟧 **2010** 🟪 **2011** 🟦

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CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Nov.'11 Prelim.	Oct.'11 Final	Nov.'10 Final
***CE* Index**	590.8	594.0	556.7
Equipment	721.0	724.7	669.0
Heat exchangers & tanks	686.6	691.5	618.3
Process machinery	674.0	674.9	627.0
Pipe, valves & fittings	899.3	906.3	847.0
Process instruments	428.0	432.5	426.1
Pumps & compressors	910.4	911.5	904.0
Electrical equipment	510.6	508.8	487.1
Structural supports & misc	767.5	769.8	688.2
Construction labor	326.9	330.0	328.8
Buildings	518.5	521.2	501.4
Engineering & supervision	330.4	330.4	336.1

Annual Index:

2003 = 402.0

2004 = 444.2

2005 = 468.2

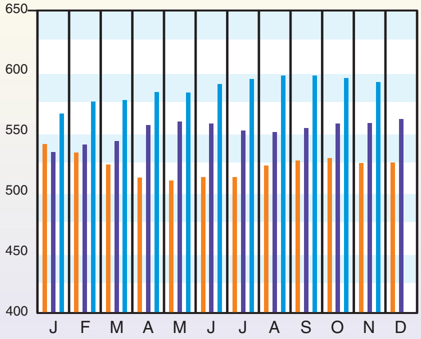
2006 = 499.6

2007 = 525.4

2008 = 575.4

2009 = 521.9

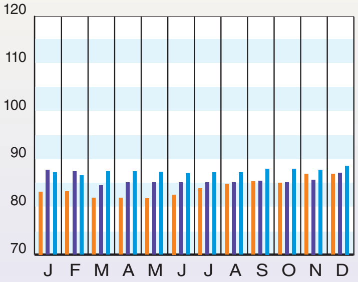
2010 = 550.8



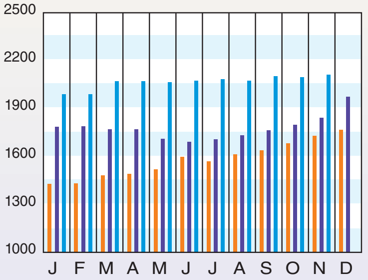
CURRENT BUSINESS INDICATORS

	LATEST			PREVIOUS						YEAR AGO		
CPI output index (2007 = 100)	Dec.'11	=	88.6	Nov.'11	=	87.7	Oct.'11	=	87.9	Dec.'10	=	87.1
CPI value of output, $ billions	Nov.'11	=	2,110.9	Oct.'11	=	2,095.8	Sep.'11	=	2,102.2	Nov.'10	=	1,843.8
CPI operating rate, %	Dec.'11	=	76.6	Nov.'11	=	75.8	Oct.'11	=	75.9	Dec.'10	=	74.9
Producer prices, industrial chemicals (1982 = 100)	Dec.'11	=	309.6	Nov.'11	=	320.2	Oct.'11	=	331.0	Dec.'10	=	285.5
Industrial Production in Manufacturing (2007=100)	Dec.'11	=	92.1	Nov.'11	=	91.3	Oct.'11	=	91.6	Dec.'10	=	88.8
Hourly earnings index, chemical & allied products (1992 = 100)	Dec.'11	=	164.8	Nov.'11	=	156.7	Oct.'11	=	157.2	Dec.'10	=	154.9
Productivity index, chemicals & allied products (1992 = 100)	Dec.'11	=	111.0	Nov.'11	=	111.0	Oct.'11	=	109.8	Dec.'10	=	113.7

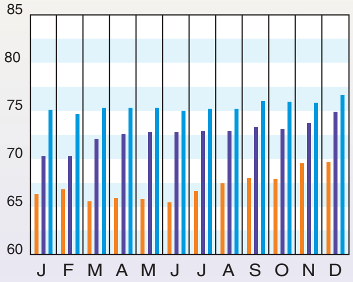
CPI OUTPUT INDEX (2007 = 100)



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



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

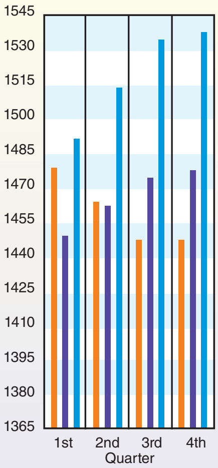
MARSHALL & SWIFT EQUIPMENT COST INDEX

(1926 = 100)	4th Q 2011	3rd Q 2011	2nd Q 2011	1st Q 2011	4th Q 2010
M & S INDEX	1,536.5	1,533.3	1,512.5	1,490.2	1,476.7
Process industries, average	1,597.7	1,592.5	1,569.0	1,549.8	1,537.0
Cement	1,596.7	1,589.3	1,568.0	1,546.6	1,532.5
Chemicals	1,565.0	1,559.8	1,537.4	1,519.8	1,507.3
Clay products	1,583.6	1,579.2	1,557.5	1,534.9	1,521.4
Glass	1,495.7	1,491.1	1,469.2	1,447.2	1,432.7
Paint	1,613.6	1,608.7	1,584.1	1,560.7	1,545.8
Paper	1,507.6	1,502.4	1,480.7	1,459.4	1,447.6
Petroleum products	1,704.9	1,698.7	1,672.0	1,652.5	1,640.4
Rubber	1,644.2	1,641.4	1,617.4	1,596.2	1,581.5
Related industries					
Electrical power	1,515.0	1,517.6	1,494.9	1,461.2	1,434.9
Mining, milling	1,659.6	1,648.6	1,623.5	1,599.7	1,579.4
Refrigeration	1,889.4	1,884.4	1,856.4	1,827.8	1,809.3
Steam power	1,574.3	1,572.2	1,546.5	1,523.0	1,506.4

Annual Index:

2003 = 1,123.6 **2004 = 1,178.5** **2005 = 1,244.5** **2006 = 1,302.3**

2007 = 1,373.3 **2008 = 1,449.3** **2009 = 1,468.6** **2010 = 1,457.4**



CURRENT TRENDS

Capital equipment prices, as reflected in the *CE* Plant Cost Index (CEPCI), decreased 0.53% from October to November. Decreases are typical during the third quarter.

Meanwhile, the CPI Operating Rate, as reported from IHS Global Insight, increased 0.8% from November to December. The CPI Output Index increased from 87.7 to 88.6 (where 2007=100).

Visit www.che.com/pci for more information and other tips on capital cost trends and methodology. ■

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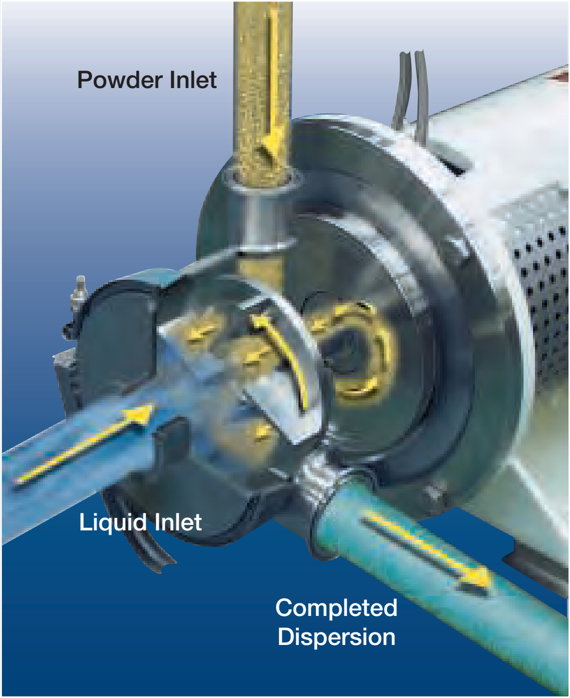

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