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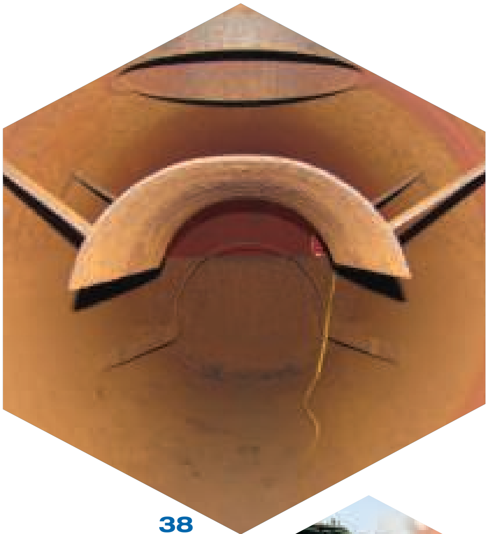
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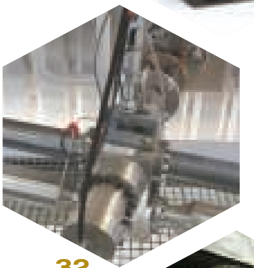

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Editor's Page

Cybersecurity: Aligning priorities

Recently, a research report titled "Intelligence Driven Cyber Defense," was issued by Ponemon Institute (Traverse City, Mich.; www.ponemon.org) with the purpose of elucidating if and how organizations were improving their ability to reduce cyber-related risks. The report, which was sponsored by Lockheed Martin (London; www.lockheedmartin.com), represents survey results of 678 U.S. information technology (IT) security practitioners, and points out a number of interesting findings.

A growing cross-industry problem

According to the report, 75% of respondents said there had been an increase in the severity of cyber attacks experienced by their organizations, and 68% said there had been an increase in frequency of these attacks. However, only 33% answered that their organizations were more effective in defending against cyber incidents than a year ago. In fact, 24% said their security posture was less effective and most (43%) said there was no change in the past year.

The respondents of the survey represent organizations in the chemical process industries (CPI; 24% were from a combination of energy, oil-and-gas, pharmaceutical and chemical sectors), financial services (21%), the Federal government (18%), healthcare (17%), utilities (16%) and other industries (4%). Cybersecurity is clearly an area of growing concern across industry sectors.

Key findings

One of the key findings summarized in the report is that the greatest cyber threat is seen as coming from inside an organization. Most respondents (37%) chose "malicious insiders" as being of more concern than potential attacks from criminal (26%) or other sources.

Another interesting finding is that while more respondents cited "user awareness" (25%) and "supply chain" (24%) as potentially having a larger impact on security than risks posed by mobile (20%) and cloud devices (18%), they said that only a small amount of funding was going to those top two areas. A total of 19% of available budget was said to be spent on user awareness and supply chain combined, whereas a disproportionate amount of the budget was allocated to mobile-device and cloud security (combined, 61% of spending).

When asked to rank the most negative consequences of a cyber attack, the survey takers cited the top five as: lost intellectual property; reputation damage; disruption to business; productivity decline and damage to critical infrastructure.

Overall, insufficient resources or budget was considered the biggest impediment to achieving a stronger cybersecurity defense, which is particularly interesting in light of the fact that responders felt the available budget was not being allocated to the areas of greatest need. The second largest barrier to better cybersecurity was said to be "insufficient visibility of people and business processes," and the third was lack of skilled personnel.

Taking the time to understand the potential areas of weakness in the complex cybersecurity arena can help organizations prioritize their resources. For example, one conclusion of this report, as one might expect, is to prioritize focus on the insider threat. ■

Dorothy Lozowski, Editor in Chief

For more on cybersecurity, see Industrial Control Systems Security: The Owner-Operator's Challenge, on www.chemengonline.com

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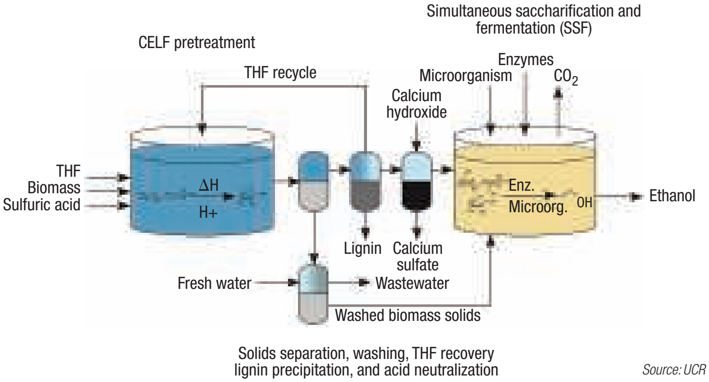
Biomass pretreatment approach can reduce enzyme requirements



Source: UCR

Biomass is a recalcitrant, heterogeneous matrix of cellulose, hemicellulose and lignin that resists microbial and enzymatic breakdown. Overcoming this recalcitrance is the major economic barrier to the conversion of biomass to sugars or other chemicals. Heat, acid and other chemicals are used in pretreatment steps to open the biomass structure so that it can be converted into fermentable sugars by enzymes.

Engineers at the University of California at Riverside (UCR; www.ucr.edu) have developed a new pretreatment method (flowsheet) that can reduce the need for enzymes in downstream steps. The technique, known as co-solvent-enhanced lignocellulosic fractionation (CELF), employs aqueous solutions of the polar, aprotic solvent tetrahydrofuran (THF) to enhance the effectiveness of the widely used dilute-acid pretreatment (flowsheet). Using CELF, a substantial portion of the fermentable sugars in hemicellulose are recovered in solution, and about 90% of the lignin is removed from the biomass, explains research leader Charles Wyman, who is a professor in the UCR Chemical and Environmental Engineering Dept. and holds the Ford Motor Company Chair in Environmental Engineering of the Center for Environmental Research and Technology in the Bourns College of Engineering at UCR. Further, the solids left after removing so much of the hemicellulose and lignin "are readily converted to glucose at enzyme loadings about one-tenth or less of those needed for conventionally pretreated biomass (such as with dilute sulfuric acid)," he adds.

"THF appears to further catalyze hydrolysis of biomass by speeding hydrolysis reactions more than competing degradation reactions, and by generally promoting the breakage of lignin bonds," says Wyman,

The research group published a paper in *ChemSusChem* describing the application of CELF to corn stover, an agricultural residue material, but Wyman says the group has also shown CELF's effectiveness with poplar and red maple hardwoods.

Making bio-based PET monomer from furfural

The research group of Yuya Tachibana at Gunma University (Gunma, Japan; greenpolymer.chem-bio.st.gunma-u.ac.jp) has developed a procedure for the production of terephthalic acid (TPA), the monomer of the widely used thermoplastic polymer polyethylene terephthalate (PET), from the inedible biomass-derived starting material furfural. Alternative, commercially available bio-based PET is made from bio-based ethylene glycol (derived from bioethanol) and petroleum-based TPA, which is made from *p*-xylene, so the amount of biomass carbon content in the PET is only 20 wt.%. This new route to TPA offers the possibility for 100% bio-based carbon in PET.

The production process consists of six steps: (1) oxidation of furfural to fumaric and maleic acids; (2) dehydration of the acids to maleic anhydride; (3) Diels-Alder (DA) reaction of anhydrous maleic acid and furan to the exo-DA adduct; (4) dehydration of the exo-DA adduct to phthalic anhydride; (5) hydrolysis of phthalic anhydride to dipotassium phthalate; and (6) transfer reaction and acidification of dipotassium phthalate to TPA. In laboratory trials, TPA was produced with 19% yield and 95–98% purity.

Tachibana says the group aims to improve the production process by reducing the number of steps to two, increasing the yield to 35–40% or even higher (50%). He says the use of bio-based TPA for plastics has the potential to fix approximately 970,000 ton/yr of CO_2 in Japan.

Edited by:
Gerald Ondrey

NANOCELLULOSE PILOT

Sappi Ltd. (Johannesburg; South Africa; www.sappi.com) will build a pilot-scale plant for low-cost nanocellulose (Cellulose NanoFibrils; CNF) production at the Brightlands Chemelot Campus (Sittard-Geleen, the Netherlands; www.brightlands.com). The pilot plant will test the manufacturing of dry re-dispersible CNF using the proprietary technology developed by Sappi and Edinburgh Napier University. The pilot plant is expected to be operational within nine months.

The pilot plant is the precursor for Sappi to consider the construction of a commercial CNF plant. Products produced using Sappi's CNF will be optimally suitable for conversion in lighter and stronger fiber-reinforced composites and plastics, in food and pharmaceutical applications, and in rheology modifiers, as well as in barrier and other paper and coating applications, says the company.

MoS_2 MONOLAYERS

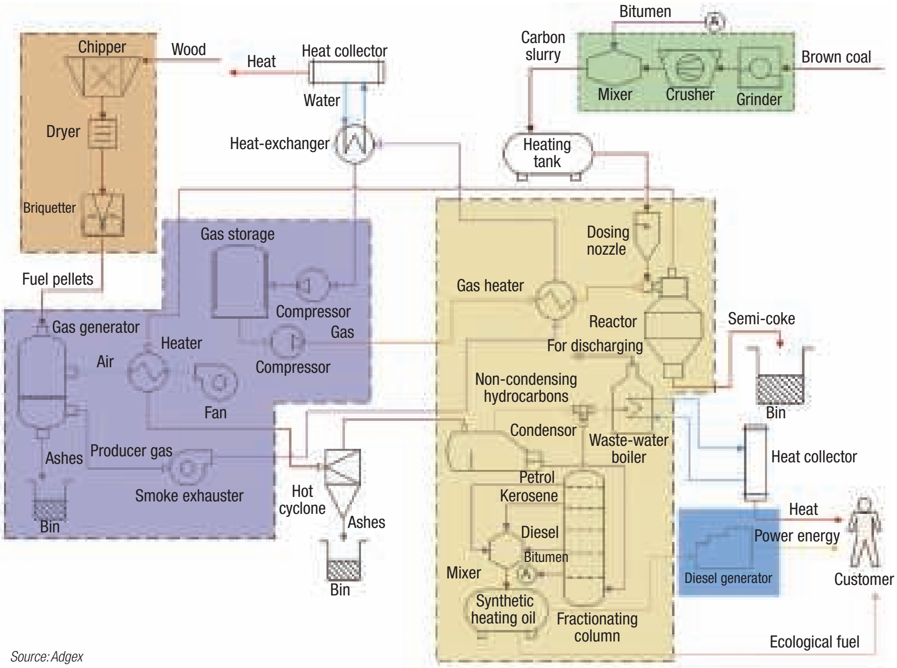
Monolayers of molybdenum disulfide (MoS_2) are attracting a lot of attention for their atomic structure, which is similar to that of the two-dimensional carbon material graphene, and for their ability to act as semiconductors in single-layer sheets. Researchers at the University of Pennsylvania (Philadelphia; www.upenn.edu) have developed a chemical vapor deposition method that results in highly crystalline flakes of monolayer MoS_2 at locations that can be precisely controlled. Using "seeds" of Mo-containing materials that are placed on a silicon substrate using conventional photolithography techniques, the pro-

(Continues on p. 13)

Note: For more information, circle the 56-digit number on p. 78, or use the website designation.

Making fuels from almost any organic material

Adgex Ltd. (Sydney, Australia; www.adgex.com) is marketing a plant, tradenamed "Green Blaze," for processing organic raw materials by means of high-speed ablative pyrolysis. According to the company, the plant (flowsheet) can be adapted to convert into synthetic fuel almost any organic material, including coal, oil wastes, municipal solid waste, industrial and commercial waste, woodchip and sawdust, animal manure and fat, food residues, sewage sludge and other carbon-containing materials. The process of high-speed ablative pyrolysis has the advantage that it allows shifting the balance toward either gaseous or liquid products by adjusting the pyrolysis temperature. The company claims its process produces far greater amounts of heat and fuel, and is far cleaner than other designs.



Source: Adgex

The process' efficiency and clean operation are achieved by careful preparation of the feedstock, by using a very small reaction area subjected to a very high rate of heat ingress and with continuous and rapid withdrawal of pyrogases from the reaction area. According to the company, combustion heat of gaseous pyrolysis products can reach up to 12.9 MJ/m^3 by virtue of the high hydrocarbon content. Output of gaseous raw material can reach 70 wt.% of dry raw material mass. Solid residue, as hydrocarbon-containing material (semi-coke) can be used as solid fuel. Calorific value of the char can reach 30 MJ/kg.

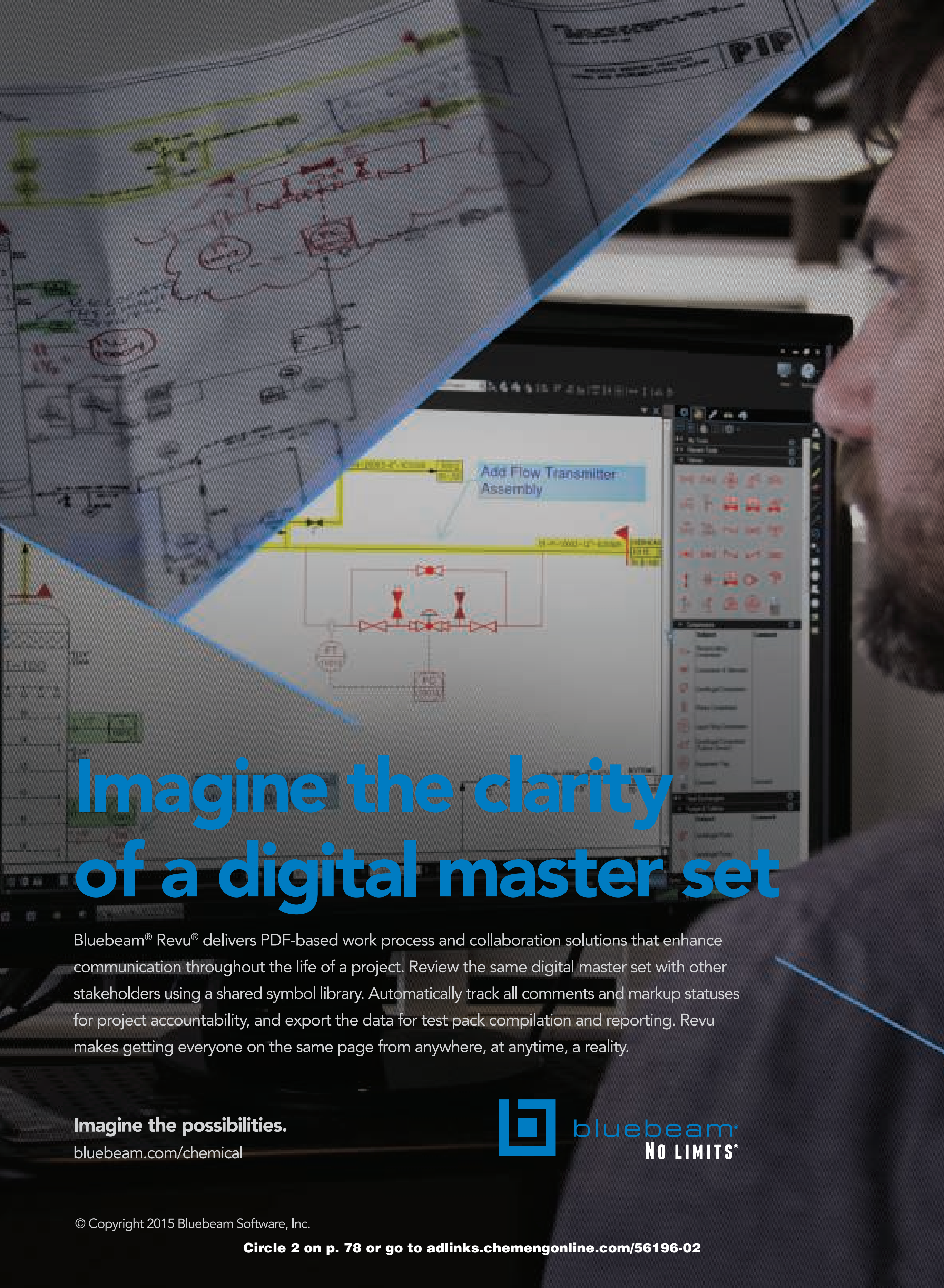
The company has tested the plant with brown coal. In this case, coal is first ground into particles of 0.5 to 1 mm size. The ground coal is then mixed with liquid pyrolysis products and exposed to additional grinding and homogenization, which generates a carbon slurry from dust-sized particles of coal with size of up to 50 µm. The generated carbon slurry is cooled and formed into 25-mm-dia. rods with a length of up to 1 m, which can be introduced into the reactor by a special ingress device. The test reactor worked for about 120 hours in the nominal temperature range of 600–650°C.

Modified MOFs could cut carbon-capture costs in half

Chemists from the University of California, Berkeley (UC Berkeley; www.berkeley.edu) have modified a metal-organic framework (MOF) compound with diamines, which enables the material to be tuned to absorb CO_2 at different temperatures, such as from air at room temperature, or fluegas from a power plant at 100°F. The absorbed CO_2 can then be released by increasing the temperature by just 50°C. In contrast, higher regeneration temperatures (80–110°C) are required for conventional aqueous liquid amines. Energy is also saved by not having to heat up the water solvent used in conventional amine-based absorbents. As a result, the modified MOFs have the potential to reduce energy consumption for regeneration by 50% or more. With these new materials, carbon-capture units on power plants could also be much smaller, making the capital costs drop "tremendously," says Jeffrey Long, a UC Berkeley professor of chemistry and faculty senior scientist at Lawrence Berkeley National Laboratory (LBNL; www.lbl.gov).

The MOFs are composites of metals (in this case, magnesium or manganese) and organic compounds that form a porous structure with microscopic, parallel channels. In a paper published last month in *Nature*, the scientists showed that the new MOFs bind CO_2 via a cooperative mechanism. Long's team found that the diamines bind to the metal atoms of the MOF and then react with CO_2 to form metal-bound ammonium carbamate species that completely line the interior channels of the MOF. At a sufficiently high pressure, one CO_2 molecule binding to an amine helps other CO_2 molecules bind next door, catalyzing a chain reaction as CO_2 polymerizes with diamine like a zipper running down the channel. Increasing the temperature by 50°C makes the reaction reverse just as quickly.

Last summer, Long co-founded a startup company, Mosaic Materials, with plans for a pilot study of CO_2 separation from power plant emissions. This would involve creating columns containing millimeter-size pellets made by compressing a crystalline powder of MOFs.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A catalyst for making aromatic hydrocarbons from biomass

A new catalyst that performs the direct and selective hydrogenolysis of the C–OH bond of substituted phenols and naphthols — two of the major components of lignin — has been developed by professor Kyoko Nozaki and colleagues at the Dept. of Chemistry and Biotechnology, University of Tokyo (Japan; www.park.itc.u-tokyo.ac.jp/nozakilab). This is said to be the first report on the selective cleavage of sp^2-hybrid C–O bonds without also cleaving the aromatic rings of lignin-derived bio-oils. The researchers say the catalyst shows promise as a way to produce basic aromatic hydrocarbons, such as benzene, toluene and xylenes (BTX) amd phenols from lignin instead of petroleum.

The catalyst, which is based on hydroxycyclopentadienyl iridium complexes, has achieved a 99% yield of the corresponding arenes, when reacting a phenol with H_2 (1 atm pressure) at 200°C. Maximum turnover numbers of 255 are also observed. Furthermore, the same catalysts were applied to the "unprecedented" selective hydrogenolysis of the sp^3-hybrid C–O bonds in aryl methyl ethers. Thus, the hydrodeoxygenation of vanillylacetone, a lignin model compound, produces alkylbenzenes as the major products via triple deoxygenation.

Scaleup for an alternative route to PC monomer

Earlier this year, Asahi Kasei Chemicals Corp. (Tokyo, Japan; www.asahi-kasei.co.jp/chemicals) has developed a new process for making diphenyl carbonate (DPC), a monomer used for making polycarbonate (PC) resins. This new route to DPC uses only an alcohol, phenol and CO_2 as feedstocks, and therefore increases the flexibility of the company's phosgene-free PC process, which uses ethylene oxide (EO) as feedstock. The company plans to build a 1,000-ton/yr validation plant at its Mizushima Works (Kurashiki, Okayama, Japan), which is scheduled to start up in January 2017, with support from Japan's New Energy and Industrial Technology Development Organization (Kawasaki, Japan; www.nedo.go.jp).

In 2002, Asahi Kasei Chemicals developed the world's first phosgene-free process for making PC, using CO_2 as feedstock (*Chem. Eng.*, December 2005, p. 16), and has since licensed five plants in the world. Now, with the introduction of this new route to DPC, there is a greater freedom in the selection of a plant location since ethylene oxide is not required.

In the new process, an alcohol and CO_2 are reacted over a new catalyst, which was developed by Asahi Kasei Chemicals, to produce dialkyl carbonate (DRC). In a second step, the DRC is reacted with phenol to give both DPC and the same alcohol that is used in the first step. Because that alcohol is recycled, only CO_2 and phenol are needed as feedstock for making DPC. With fewer process steps, the new process is also more energy efficient, enabling production costs to be reduced substantially, says the company.

Commercial-scale production for bio-based levulinic acid

This summer, GFBiochemicals Green Future S.r.l. (Milan, Italy; www.gfbiochemicals.com) will start commercial-scale production of levulinic acid using its proprietary, continuous-production process. The company says it will produce 2,000 metric tons per year (m.t./yr) at its plant in Caserta, Italy later this year, followed by scaleup to 4,000 m.t./yr in 2016 and then 8,000 m.t./yr by 2017. Initially, the feedtock for the production will be starch, but the company plans to switch to cellulose feedstock during 2016.

Recognized by the U.S. Dept. of Energy (Washington, D.C.; www.energy.gov) as one of the twelve future bio-based building-block chemicals, levulinic acid has potential as a starting material for a wide number of compounds, with applications in pharmaceuticals, agrochemicals, flavors and fragrances and food additives, as well as resins and coatings, plasticizers, solvents and fuel additives.

A patented process makes stabilized proteins

XstalBio Ltd. (Glasgow, U.K.; www.xstalbio.com) has recently been issued a patent (US 8,932,715) covering the use of precipitation-stabilizing additives for the manufacture of dry powders of therapeutic proteins, including monoclonal antibodies (mAbs). Compared to lyophilization, the XstalBio precipitation process offers advantages of speed, cost and dose-flexibility and it produces humidity- and temperature-stable powders that are much easier to handle than spray dried particles, says the company.

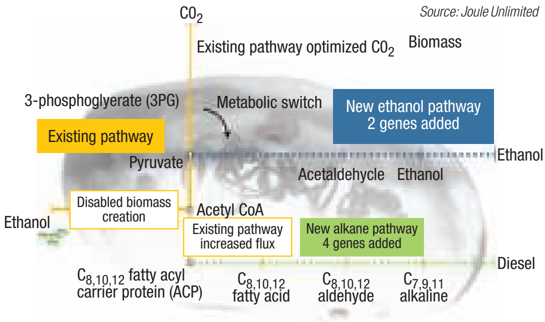
Applications in development include proven multiyear (more than 7 yr) intermediate storage of protein drugs as bulk dry powders, and production of high-concentration mAb solutions suitable for subcutaneous injection. The proprietary technology allows delicate protein drugs, unstable in aqueous solution, to be rapidly and cost-effectively precipitated into very stable dry microparticles with full retention of bioactivity. XstalBio has exclusive rights to the patented technology and is developing a commercial process suitable for good manufacturing practice (GMP) manufacture of metric-ton-per-annum quantities of protein powder.

Lignin to adipic acid

A new study from the National Renewable Energy Laboratory (NREL; Golden, Colo.; www.nrel.gov) demonstrates the conversion of lignin-derived compounds to adipic acid, an important precursor for making nylon and other chemicals. The new route offers a “greener” alternative to conventional methods (oxidation of cyclohexanol and cyclohexone with nitric acid), which generate nitrous oxide — a greenhouse gas.

Recently published in *Energy & Environmental Science*, the research demonstrates how lignin-derived compounds can first be converted to muconic acid via a biological process. Muconic acid can then be separated from the biological culture and catalytically converted into adipic acid. A patent application has been filed on this research.

End-to-end demonstration of CO_2-to-ethanol process achieved



Joule Unlimited (Bedford, Mass.; www.jouleunlimited.com) announced recently that it has achieved end-to-end production of ethanol from carbon dioxide and sunlight, using its specially designed photobioreactor and engineered cyanobacteria. The full production runs occurred late last year at the company's Hobbs, N.M. facility and ethanol from the process is undergoing further testing by German automaker Audi, a strategic partner of Joule.

Joule is planning to construct a larger-scale production plant that will produce 15 million gal/yr of ethanol from CO_2 and will break ground in 2017. The facility may also produce a C11 molecule for diesel fuel that is currently also under development by the company.

In 2009, Joule initially demonstrated its "reverse combustion" process (diagram), in which strains of engineered cyanobacteria metabolize CO_2 in the presence of sunlight and brackish water to produce ethanol and diesel, depending on the strain used.

Over the past year, Joule has improved its process, both on the biocatalyst side and the process engineering side, explains David St. Angelo, senior vice president for engineering at Joule. Strain selection and further engineering of the cyanobacteria has improved the performance of the biocatalyst, St. Angelo says, and the company is doubling the size of its photobioreactor capsule, from 50 to 100 m in length. "The longer reactor is part of an effort to make a lower cost, robust continuous process. The capsule has been designed for a lower pressure drop across the reactor, and hence, lowered pumping costs."

cess can produce flakes of high-quality MoS_2 material at predetermined locations, in contrast to existing techniques where the flakes are distributed randomly, and are of lower quality. The researchers hope the method will help allow future MoS_2-based integrated circuitry and sensors.

SELF-CLEANING PAINT

Researchers at University College London (UCL; U.K.; www.ucl.ac.uk) and Dalian University of Technology (China;http://en.dlut.edu.cn/) have developed a new paint that makes robust self-cleaning surfaces. The coating can be applied to clothes, paper, glass and steel, and when combined with adhesives, maintains its self-cleaning properties after being wiped, scratched with a knife and scuffed with sandpaper.

The study, published last month in *Science*, shows how the new paint made

(Continues on p. 14)

from coated titanium dioxide nanoparticles can give a wide-range of materials self-cleaning properties, even during and after immersion in oil and following damage to the surface. Different coating methods were used to create the water-repellent surfaces, depending on the material. A spray-gun was used to coat glass and steel, dip-coating for cotton wool and a syringe to apply the paint onto paper.

TILTING TREES HELPS

Willow trees growing at an angle of 45 deg respond by producing a sugar-rich gelatinous fiber, making up to five times more sugar than when grown normally. This is the conclusion — important for boosting biofuel yields — of researchers from Imperial College London (U.K.; www.imperial.ac.uk), who used X-ray micro-computed tomography (CT) to study the growing plants. ❑

MOF material improves CO_2-capture effectiveness

Industrial CO_2 capture is currently done by absorption in liquid amines, such as monoethanolamine. However, absorption-based carbon capture carries a significant energy penalty for regenerating CO_2 in a stripping column — power plants can lose 20–30% of their energy output by capturing CO_2 with conventional technology.

Researchers at New Mexico State Univ. (NMSU; Las Cruces; www.nmsu.edu) have invented novel metal-organic-framework (MOF) materials that selectively capture CO_2 while cutting the energy penalty in half. Known as zeolitic imidazolate frameworks (ZIFs), the materials have surface areas an order of magnitude higher than zeolites, and considerable thermal and chemical stability, explains project lead Nasser Khazeni, a Ph.D. candidate.

Khazeni synthesized a new subgroup of ZIFs that incorporates a ring carbonyl group in its organic counterpart. These materials separate CO_2 from gas mixtures more selectively and have higher CO_2-uptake capacities compared to similar structures, Nasser says.

In an industrial setting, the ZIF would be granulated and packed into an adsorption column, through which exhaust would pass, Nasser says. NMSU has filed for a patent and is seeking to license the invention. Industry sectors have shown interest, Nasser notes.

A new primary aluminum process to be piloted

Last month, Hydro ASA (www.hydro.com) entered into a contract with Multiconsult (both Oslo, Norway; www.multionsult.no) for engineering services for Hydro's planned pilot plant at Karmøy, Norway. The Karmøy pilot aims to verify what Hydro calls the world's most energy- and climate-efficient electrolysis technology for aluminum production. The process being piloted is targeting a 15% reduction in energy consumption per kilogram of aluminum produced compared to the world average, says the company.

The full-scale pilot plant — costing an estimated NOK3.9 billion ($0.5 billion) — is planned to have a production capacity of 75,000 m.t./yr, with startup in the second half of 2017 at the earliest. The Multiconsult contract covers the engineering design of demolition work, a new potroom, rectifier and transformer substations, power distribution, infrastructure and support systems. ■

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Plant Watch

Arkema to expand production of PEKK specialty polymer in the U.S. and France

March 9, 2015 — Arkema (Colombes, France; www.arkema.com) plans to double production capacity for poly-ether-ketone-ketone (PEKK) polymer in France in the first half of 2016. Furthermore, the company plans to build a PEKK-production plant on its Mobile, Ala. site, which would be scheduled for startup in the second half of 2018.

Perstorp starts up plasticizer plant extension in Sweden

March 5, 2015 — Perstorp Holding AB (Malmö, Sweden; www.perstorp.com) has successfully started up a new plant that produces key raw materials for the general-purpose PVC plasticizers, valeraldehyde and 2-propylheptanol, and associated chemicals at the company's site in Stenugsund, Sweden. With this investment, Perstorp has boosted the site's capacity by 150,000 metric tons per year (m.t./yr).

BASF expands its capacity for specialty amines in Ludwigshafen

March 4, 2015 — BASF SE (Ludwigshafen, Germany; www.basf.com) is significantly expanding its production capacity for about 20 specialty amines at its site in Ludwigshafen. The expanded facilities are planned to go onstream gradually by early 2017. The specialty amines are used for the manufacture of coatings, lubricants, crop-protection products and pharmaceuticals.

SNC-Lavalin awarded fluegas-treatment contract in Romania

March 3, 2015 — SNC-Lavalin (Montreal, Que., Canada; www.snclavalin.com) has been awarded a contract for a new gas-emissions treatment facility at a nitrogen, phosphorus and potassium (NPK) fertilizer production plant in Târgu Mures, Romania. The facility will treat an estimated 183,000 Nm^3/h of air from the NPK plant, which is expected to be fully operational in early 2016.

Wacker builds new production plant for specialty polymers

March 3, 2015 — Wacker Chemie AG (Munich, Germany; www.wacker.com) is building a new €8-million plant for specialty monomers with a production capacity of 3,800 m.t./yr at its Burghausen, Germany site. The plant is scheduled for startup in the second quarter of 2015.

ABB to build manufacturing site in Tennessee

March 3, 2015 — ABB (Zurich, Switzerland; www.abb.com) will invest $30 million in building a new manufacturing site near Memphis, Tenn. The new facility will assemble products from across ABB's low-voltage products portfolio, including breakers, switches and modular enclosures.

AkzoNobel breaks ground on alkoxylation facility in Ningbo

March 2, 2015 — AkzoNobel (Amsterdam, the Netherlands; www.akzonobel.com) recently broke ground on a new alkoxylation facility at its site in Ningbo, China, bringing the company's total investment in Ningbo to more than €400 million. This new facility will increase capacity by nearly 18,000 m.t./yr.

Solvay to build specialty polymers PEEK-production unit in U.S.

February 24, 2015 — Solvay S.A. (Brussels, Belgium; www.solvay.com) is building a new unit at its Augusta, Ga. site to expand its production capacity of polyether etherketone (PEEK). The new unit is expected to come onstream in mid-2016 and, combined with an expansion already underway in Panoli, India, will raise Solvay's total PEEK-resin production capacity to more than 2,500 m.t./yr.

ThyssenKrupp Industrial Solution wins order for Hungarian nitric-acid plant

February 20, 2015 — ThyssenKrupp Industrial Solutions AG (Essen, Germany; www.thyssenkrupp-industrial-solutions.com) has won a contract from fertilizer manufacturer Nitrogénmüvek Zrt for a nitric acid plant to be built at Pétfürdö, Hungary. The plant's production capacity will be 1,150 m.t./d nitric acid, and completion is scheduled for 2017.

Yara and BASF to build ammonia plant in Freeport

February 19, 2015 — Yara International (Oslo, Norway; www.yara.com) and BASF will build an ammonia plant at BASF's site in Freeport, Tex., which will be owned 68% by Yara and 32% by BASF. The total capital investment for the plant, which will have a capacity of 750,000 m.t./yr, is estimated at $600 million.

Honeywell to provide control-systems support for Sasol coal-to-liquids plant

February 17, 2015 — Honeywell (Morristown, N.J.; www.honeywell.com) will provide comprehensive services and support for the control systems of what is said to be the world's largest coal-to-liquids (CTL) plant, Sasol's synthetic fuels and chemicals complex in Secunda, South Africa.

Mergers & Acquisitions

Evonik to acquire Monarch Catalysts

March 11, 2015 — Evonik Industries AG (Essen, Germany; www.evonik.com) has signed an agreement with India-based Monarch Catalyst

Pvt. Ltd. to acquire 100% of the company's shares. The transaction is expected to close during the first half of 2015.

Alcoa expands titanium, specialty offerings with RTI acquisition

March 10, 2015 — Alcoa Inc. (Pittsburgh, Pa.; www.alcoa.com) has signed a definitive agreement to acquire RTI International Metals, Inc., a supplier of titanium and specialty-metal products and services for the aerospace, defense, energy and medical-device markets. The transaction has an enterprise value of $1.5 billion.

Data analytics company Wood Mackenzie acquired by Verisk

March 10, 2015 — Data analytics provider Verisk Analytics, Inc. (Jersey City, N.J.; www.verisk.com) has signed an agreement to acquire Wood Mackenzie (Edinburgh, Scotland; www.woodmac.com), an analytics provider for the chemical, energy and mining sectors, for around $2.8 billion.

Sonoco acquires majority stake in Brazilian packaging firm

March 9, 2015 — Sonoco Products Co. (Hartsville, S.C.; www.sonoco.com), announced the signing of a definitive agreement to acquire a majority interest in Graffo Paranaense de Embalagens S/A (Graffo), a flexible packaging business located in Pinhais, Curitiba, Brazil. The transaction is expected to close in the second quarter of 2015.

Total acquires majority stake in plastics company Polyblend

March 5, 2015 — Total S.A. (Paris, France; www.total.com) has acquired a majority 68% interest in Polyblend GmbH (www.polyblend.de; Bad Sobernheim, Germany), which manufactures plastics for the automotive industry. The transaction is aligned with Total's strategy to develop higher-value polymers.

Bayer MaterialScience acquires composite materials specialist

March 2, 2015 — Bayer MaterialScience AG (Leverkusen, Germany; www.materialscience.bayer.com) concluded the takeover of Thermoplast Composite GmbH (TCG; Langenfeld, Germany; www.thermoplast-composite.com), a producer of thermoplastic-fiber composites. Bayer MaterialScience plans to expand TCG's production capacity in various locations, beginning in the Nuremberg metropolitan region.

Polypore business segments divested to Asahi Kasai and 3M

February 23, 2015 — Asahi Kasei Chemicals Corp. (Tokyo, Japan; www.asahi-kasei.co.jp) has entered into an agreement to acquire the Energy Storage business of Polypore International, Inc. (Charlotte, N.C.; www.polypore.net) for approximately $2.2 billion. In conjunction, Polypore also divested its Separations Media segment to 3M (St. Paul, Minn.; www.3m.com) for around $1 billion.

Mann+Hummel takes full ownership of Microdyn-Nadir

February 17, 2015 — Microdyn-Nadir GmbH (Wiesbaden, Germany; www.microdyn-nadir.de), a membrane and module manufacturer, is now a 100% subsidiary of Mann+Hummel GmbH (Ludwigsburg, Germany; www.mann-hummel.com). With this transaction, Mann+Hummel increases its Microdyn-Nadir stake from 50 to 100%. ■

Mary Page Bailey

New Frontiers in Metals Recycling

Precious and rare-earth metals are used in countless consumer products, and advanced processes to efficiently recover these materials from waste present promising alternative sources

Umicore

FIGURE 1. Electronics waste — including discarded circuit boards and mobile phones — can contain many valuable materials

IN BRIEF

TREATING UNIQUE PRECIOUS METALS

PGMS FROM AUTOCATALYSTS

SAFER STRIPPING

RARE-EARTH METALS

COMMERCIAL FUTURE

Metal is all around us — we drink out of aluminum cans and eat with stainless-steel (or perhaps even silver) silverware. But we may not give much thought to the platinum stored within our vehicles or the europium contained in our smartphone screens. We also may not consider what happens to these materials when they reach the end of their useful life. Potentially valuable product streams can be obtained if the precious and rare-earth metals contained in these products are efficiently recovered. This article will examine some advanced techniques for recycling a variety of consumer products to recover precious metals, as well as the more elusive rare-earth metals.

Treating unique precious metals

Precious metals are crucial in many diverse products and applications, and determining the most efficient recycling processes for each material presents challenges. Umicore N.V. (Brussels, Belgium; www.umicore.com) operates a complex metals-recycling facility in Hoboken, Belgium, that treats approximately 350,000 metric tons per year (m.t./yr) of industrial residues and other recyclable materials, including electronics waste (Figure 1), such as mobile phones and printed circuit boards (PCBs), and automotive and industrial catalysts. The recycling process is unique, says Umicore communications and general services manager Marjolein Scheers, because of the variety of materials handled and recovered — the process recovers 17 different metals, including precious metals, such as gold, silver and the platinum-group metals (PGMs) platinum, palladium and rhodium.

"The operation is designed so that raw materials enter the process at the most optimal step," explains Scheers. This is determined by the individual material's physical properties, analytical fingerprint and recovered value. The process begins with a smelter, where precious metals are separated from other metals. The smelter furnace applies a submerged-lance-combustion technology, which involves the injection of oxygen-enriched air into a molten-metal bath. Says Scheers: "Umicore is the only company to apply this technology on such a large scale with such a variety of materials." Following the smelter are copper-leaching and electrowinning steps, after which the precious metals are collected in a residue that is further processed at an in-house refinery. According to the company, on average, over 95% of the metals contained in the feed can be recovered.

Umicore understands the complexity of recycling waste electrical and electronic equipment (WEEE), and in February of this year, received compliance certification by two European trade organizations (Eurometaux and the European Electronics Recyclers Association) for WEEE recycling. "As the recycling of WEEE wastes consists of various steps, such as collection, pre-processing and end-processing, it is logical that the efficiency and performance of the entire chain is monitored and evaluated," says Scheers.

PGMs from autocatalysts

One major application for precious PGMs is in the catalytic converters in automobiles. When an automobile has reached the end of its use-

BASF

FIGURE 2. Spent automotive catalysts contain valuable precious metals, such as palladium, platinum and rhodium

ful life, the PGMs contained within the catalytic converter can be recycled. BASF SE (Ludwigshafen, Germany; www.basf.com) operates three facilities worldwide that recover PGMs (platinum, palladium and rhodium) from spent automobile catalysts (Figure 2) in either ceramic or metallic converter systems. BASF's process for recovering PGMs from ceramic converter systems is shown in Figure 3. Ceramics represent a larger-volume market than metallic converters, and a recent expansion project at the company's Cinderford, U.K. site more than doubled the site's processing capacity for ceramic converters. The upgrades at Cinderford, which went onstream in February of this year, involved the installation of new milling and de-canning equipment.

A variety of factors drives demand for recovered PGMs. The recovered metals are used to supply BASF's Mobile Emissions Catalysts business, which, says the company, means that they are then used in a new generation of emissions-reduction catalysts. According to BASF, demand for these catalysts has increased in line with automotive production, along with "new and more restrictive automotive emissions regulations around the world." In addition to the Cinderford location, BASF operates two autocatalyst-recycling facilities in the U.S.The benefits of large-scale PGM recycling are evident, says the company, citing "the lower cost of recycling processes versus other metal sources (mining or open-market purchase), as well as the sustainability and environmental benefits of recovering metals from end-of-life materials that would otherwise end up in landfills."

BASF

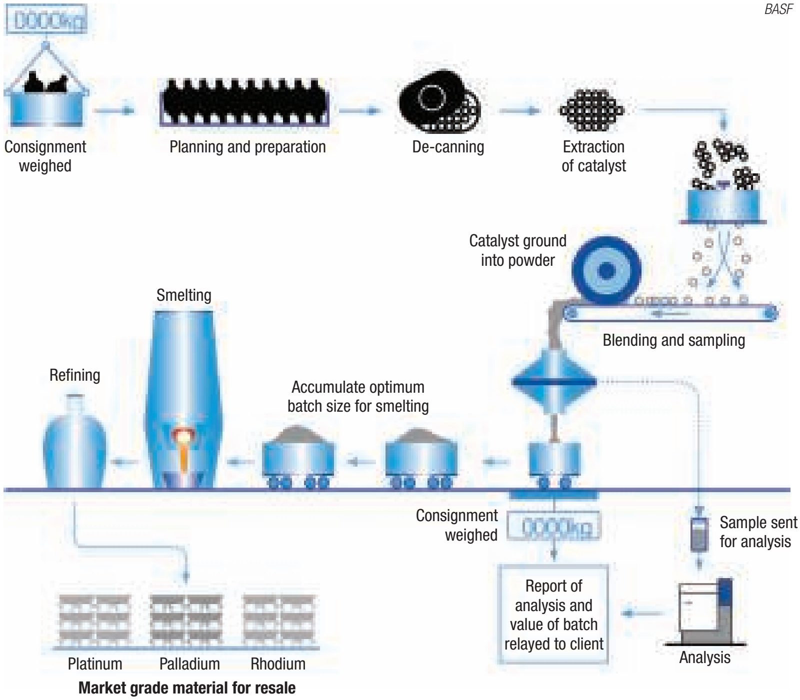

FIGURE 3. Recently expanded at BASF's Cinderford site, this process recycles platinum-group metals from automotive catalysts for use in new mobile-emissions catalysts

Safer stripping

Electronics components, specifically PCBs, represent a very promising area for the recovery of precious metals, as gold can be stripped from PCBs and recycled. Typical industrial gold-stripping processes utilize cyanide or aqua regia (nitro-hydrochloric acid) solutions. UWin Nanotech Co. (New Taipei City, Taiwan; www.uwin-nano.com) has recently patented what is said to be more environmentally friendly, less toxic gold-stripping chemicals — UW-700 (sulfide solution) and UW-860 (citrate solution). "Aqua regia is a highly corrosive solution, and cyanide is a very toxic solution," UWin managing director Kenny Hsu explains, "whereas UW-700 is a neutral gold-stripping solution, and is also highly selective." For UW-860, Hsu elaborates, saying that the solution "is different from aqua regia because it doesn't damage the plastic, ceramic, silicon, titanium or stainless-steel substrate."

After treatment with UW-700, the gold is completely stripped from the PCB, while the remaining materials are left intact (Figure 4). The recovered gold is isolated and can be re-sold for use in optical-electrical, plating and decorative applications. According to the company, applying this technology to 1 m.t. of PCB waste from cellular phones can result in the extraction of 400 g of gold.

Using these chemicals, the company has developed modular, automatic gold-stripping machines (Figure 5), which have deployed commercially worldwide, including locations in Taiwan and Texas. The company is also collaborating with a mechanical manufacturer to develop new types of stripping machines. UWin has also developed technologies for selective stripping of other precious metals, including silver, palladium and indium. In January 2015, the company released UW-195, a cyanide-free platinum-stripping agent that is compatible with stainless-steel, plastic or ceramic substrates.

Rare-earth metals

Compared to their precious-metal counterparts, and despite a growing demand, commercial recycling and recovery techniques remain elusive for the 17 rare-earth metals (REMs). China, in recent years, has become the world's most significant source of REMs, but recent regulatory changes may lead to a global shortage, forcing manufacturers to seek new methods to obtain REMs, including recycling.

"Rare-earth elements really are not scarce in the world. You may find some of them in your own backyard," UWin

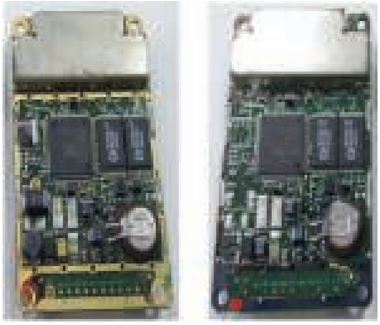

FIGURE 4. A comparison of printed circuit boards before (left) and after (right) stripping treatment illustrates that the gold is removed while the remaining materials remain intact

UWin

FIGURE 5. Automatic gold-stripping machines allow for efficient treatment of printed curcuit boards

explains Eric Peterson of the Idaho National Laboratory (INL; Idaho Falls, Idaho; www.inl.gov). "However, it is rare to find them in locations where they occur in reasonably high concentrations that allow for economical mining of the materials."

INL is part of the Critical Materials Institute (CMI; www.cmi.ameslab.gov), an Energy Innovation Hub sponsored by the U.S. Dept. of Energy through the Advanced Manufacturing Office. Comprised of national laboratories, universities and companies at the forefront of REM research, CMI brings a four-tiered approach to tackling advanced rare-earth projects, focusing on the development of more efficient separations, recovery and recycling processes, and alternative materials that can potentially serve as REM substitutes, as well as bringing end-of-life awareness to manufacturers whose products utilize rare-earth metals.

While commercial technologies do exist for recovering REMs, these processes have some shortcomings. "There are many technological approaches that may make it possible to recover and recycle the elements," says Peterson. "However, due to the specific chemistries of each of the rare-earth elements, it is necessary to develop approaches to recycle the elements independently." Current commercial technologies may result in complex mixtures, which require further purification before yielding a usable product. The economy of these technologies is also highly dependent on the market value for the recovered metals, as in the case of recycling cerium from automotive catalysts or fluidized catalytic cracking (FCC) catalysts.

CMI is developing a number of processes for recovering REMs from various sources, including electronics waste, phosphor powders from fluorescent lighting and LEDs and permanent magnets from computer disc drives and wind turbines. "Phosphors are moving very quickly to the commercial scale," says Peterson of CMI's phosphor-recovery projects, which target fluorescent lamps as the source of phosphor powder. Phosphor powder can contain 5–25% by weight rare-earth elements, such as europium or terbium, in a cerium-, yttrium- or lanthanum-phosphate matrix.

In one CMI project, a supercritical-fluids process selectively extracts rare-earth metals from the phosphor powders, which also contain mercury. "The economics of the process are aided by the recovery and isolation of the mercury that is also contained in the phosphor powder, plus the recovery of the glass, aluminum and steel," INL's Peterson explains. In another project, CMI's main focus is concentrating the rare-earth oxides (REOs) in phosphor dust and separating out the non-REO materials via hydrometallurgical methods.

Perhaps the most promising REM-recovery process CMI has developed involves membrane solvent extraction. CMI has demonstrated and is licensing the process, and expects to see full-scale realization of the project in 2.5 years. This extraction process sets itself apart from other rare-earth recovery techniques in that a more dilute stream can be used for extraction. A dispersion-free, supported, liquid-membrane solvent-extraction process separates and recovers REMs, such as yttrium, europium, praseodymium and neodymium, from permanent magnets and halophosphate phosphors. Operating under non-equilibrium conditions, the extraction process overcomes the stability issues arising due to the gradual loss of extractant in normal solvent-extraction processes. Other REM-recovery projects in development at CMI include recovery of indium and REOs from thin-film plasma display panels, recovery of overspray from coating processes, bioleaching using microorganisms and biosorption in an aqueous solution.

Since most of their processes utilize components of discarded consumer products, CMI recognizes that the ability to readily recover and recycle REMs begins with the initial manufacture of the product. Involving manufacturers, and emphasizing the importance of end-of-life considerations in manufacturing processes, eases the complexities of recycling these materials. For instance, if disc drives are manufactured so that the magnets are easily removable, recycling the REMs contained within becomes a much less labor-intensive task. This will benefit the manufac-

"Rare-earth elements really are not scarce in the world. You may find some of them in your own backyard," explains Eric Peterson of the Idaho National Laboratory

turers, as well, through the increased availability of recycled REMs, while decreasing the dependence on virgin materials.

Another group working to develop recycling processes for REMs is Sintef (Trondheim, Norway; www.sintef.no), as part of the "Value from Waste" consortium formed by various European research and technology organizations. Sintef's work has targeted two main sources for REM recovery — nickel metal-hydride (NiMH) batteries and permanent magnets that contain neodymium (Nd) — focusing mainly on the use of high-temperature electrolysis for the direct extraction of REM alloys from magnets. Says Sintef senior research scientist Ana Maria Martinez, "This can be considered a direct electro-refining process, as it allows the extraction of a rare-earth alloy that can be used in the manufacture of new permanent magnets directly from the waste material." Figure 6 shows a simplified model of the magnet-recycling process, which begins with the scrap material being placed into an anode compartment. Here, the REMs present in the magnets (typically Nd, but also dysprosium, praseodymium or terbium) are anodically dissolved in the form of rare-earth ions, which will deposit at the cathode as rare-earth alloys. Other elements present (such as transition metals) will remain at the anode as sludge.

Sintef

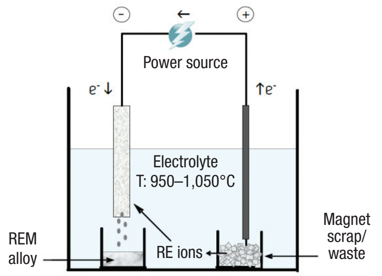

FIGURE 6. Rare-earth metals contained in permanent magnets can be recovered via high-temperature electrolysis

"At the moment, the high-temperature electrolysis approach has only been applied in the case of Nd-based permanent magnets," comments Martinez, "but the method could be suited to any conductive rare-earth-containing material." The technology was recently submitted to the European Commission to receive economic support for demonstration in industrial environments. In addition to end-of-life Nd-containing magnets, the recycling process can be supplemented with the use of scrap from the manufacture of new magnets. According to Martinez, "This is seen as the most practical solution to start operating recycling plants effectively."

Sintef's process for recovering REMs from NiMH batteries involves pyrometallurgical refining. "This high-temperature approach is able to completely separate the valuable nickel-based alloys and REO concentrates from the negative electrodes of NiMH batteries," says Martinez. The output material is subsequently fed into a high-temperature electrolysis process to produce a mixture of REMs, which can be used in new battery electrodes. As with Sintef's magnet-recycling process, the next steps will be to extrapolate these technologies to other REM-containing waste streams.

Like CMI, Sintef recognizes the challenges associated with rare-earth recycling, emphasizing the importance of implementing efficient systems for obtaining usable REM-containing scrap. “The process of separating and collecting the magnets safely from end-of-life products not only requires a great deal of time and effort, but also uses acids and other chemicals,” says Martinez. “This results in toxic liquid wastes, the disposal of which creates environmental and cost issues.” Once again, attention is brought to the limitations that initial manufacturing considerations can put on recycling.

Commercial future

On the commercial front, some companies are developing REM-recycling processes. In France, Solvay S.A. (Brussels, Belgium; www.solvay.com) has demonstrated a process for recycling phosphor powders (containing lanthanum, cerium, terbium, yttrium, europium and gadolinium) from fluorescent light bulbs and lamps. At the company’s St. Fons site, the phosphor powders are separated from glass and other components and suspended in an aqueous solution. In solution, the phosphor powders undergo a chemical reaction, further concentrating the REMs. After separating out the liquid and drying, the remaining REM-concentrate powder is then sent to another site, La Rochelle, which specializes in rare-earth purification.

At the 2014 Minerals, Metals and Materials Society Annual Meeting & Exhibition, Hitachi Ltd., (Tokyo, Japan; www.hitachi.com), through the company’s Yokohama Research Laboratory, announced a recycling process for permanent magnets that includes steps for the disassembly of scrap products, followed by pyrometallurgical recovery of neodymium and dysprosium using molten magnesium as an extraction medium. It was reported that the company can process several kilograms of magnets per day using this technology.

Mining companies, as well, are acknowledging the importance of REM conservation, and are looking into technologies to recover and recycle REMs from mining waste. In March of this year, Ucore Rare Metals Inc. (Bedford, Nova Scotia, Canada; www.ucore.com) secured exclusive rights to advanced molecular-recognition technology that can be used for REM separation and recycling. According to the company, this technology can recover more than 99% of the REM content in a stream. In February, Pele Mountain Resources (Toronto, Ont., Canada; www.pelemountain.com) announced a plan for the dedicated recycling of the REM-containing mineral monazite, from which neodymium and praseodymium, along with smaller amounts of other REMs, can be produced.

It seems that across the board, from miners to electronics manufacturers to consumers, the importance of conserving rare-earth materials, as well as precious metals, is clear. Although recycling processes for REMs have not yet reached the level of efficiency and commercial feasibility of other metals-recycling techniques, the great deal of development work in the area is certainly promising. ■

Mary Page Bailey

The Changing Face of Gas Analysis

Calibration-free, modular and automated gas analyzers offer benefits to the chemical process industries

IN BRIEF

REDUCED MAINTENANCE AND CALIBRATION

MODULAR SYSTEMS

AUTOMATION

FIGURE 1. The TDLS200 with TruePeak was designed to make fast, accurate measurements on near-infrared absorbing gases in harsh process environments

Yokogawa Corp. of America

While there are countless types of gas analysis instruments available, users of every type are asking for technologies that require less of their time. Processors are demanding instruments that require minimal calibration and maintenance, as well as units that are modular and capable of self diagnostics and other automated functions to save labor and plant resources. Fortunately, providers of analytical devices are meeting these needs by changing the way gas measurement is addressed in the chemical process industries (CPI).

Reduced maintenance and calibration

"When we first launched products into the gas-analysis space, we began by speaking to our processing customers and found that running and maintaining gas analysis instruments, in an effort to get proper performance and fulfill safety requirements, was absorbing a lot of resources in chemical and petrochemical plants," says Jean-Nicolas Adami, manager of business development for gas analytics, Mettler-Toledo AG, Process Analytics (Urdorf, Switzerland; www.mt.com). "End users were spending a lot of time on maintenance and preemptive tasks, such as calibration of instruments."

In addition, Adami says, many processors were taking extractive measurements, meaning a sample of gas is taken from the process, conditioned and finally analyzed. "This is often done to protect the instruments from dust, gas, corrosion, condensation and other harsh conditions," he explains. "However, there's a lot of time spent maintaining such a process and system."

Jesse Underwood, product manager for TDLS analyzers with Yokogawa Corp. of America (Sugar Land, Tex.; www.yokogawa.com/us), agrees. "Any time you can eliminate sample extraction, sample transport and sample systems, you reduce time spent on analysis and eliminate failure points in the overall analysis system," he says.

For this reason, both Mettler-Toldeo and Yokogawa provide in-situ tunable diode laser (TDL) spectrometers, in addition to extractive TDL systems, as an alternative to traditional instruments for single-gas measurement in the CPI.

TDL analysis technology offers "significant" benefits to end users, suggests Adami. Most notably, "they [TDL spectrometers] don't suffer from instrument drift, which is common for just about every type of technology," he says. "Using a tunable diode laser provides the ability to lock onto an emission frequency so there's no chance for drift. In addition, the high selectivity of

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Mettler-Toledo

FIGURE 2. The GPro 500 TDL series offers interference-free, drift-free TDL technology along with a folded optical path principle, which simplifies alignment

the TDL instrument means there's no reason what-so-ever to perform calibration if installed properly in stable process conditions," explains Adami. (Although instruments must still be verified in accordance with applicable regulations.)

And, when used to replace extractive sampling systems, the reduction in hands-on maintenance time is even greater, says Underwood. "Typical extraction systems require a minimum of monthly preventive maintenance, while in-situ systems require maintenance on demand, meaning there is no preventive maintenance for tasks such as changing filters and traps in a sampling system," says Underwood.

Another perk of this technology is that because in-situ analysis is made directly inside the process pipe, it is more accurate than extractive sampling. "Think of all the changes that can happen when the gas goes through the sampling system," says Adami. "For example, you have to extract moisture from the gas, which may change the concentration of the gas, which, in turn, means processors can't be 100% sure that the measured value is representative of what's really going on inside the process."

In addition, Underwood says TDL analysis technology is safer in many potentially explosive applications. "Most analytical techniques require that the sensor be exposed to the process, which can be a source of potential hazard once those are heated during the process. But, because it employs a non-contacting sensor, a TDL-based measurement device does not provide an ignition source," he says.

To bring these benefits to processors, Yokogawa offers the In-Situ Tunable Diode Laser Spectroscopy (TDLS200) with TruePeak technology (Figure 1), which is a technique that allows measurements to be taken with direct absorption, making the device immune to background gas interference, says Underwood. It was designed to make fast, accurate measurements on near-infrared (NIR) absorbing gases in harsh process environments, where conditions may include high temperature or pressure and corrosive, aggressive and high-particulate-content materials. It is suitable for applications such as combustion control; monitoring of CO, CH_4 and moisture to enable burner flame-out; measurement of O_2 on flare lines, alkylation units and gas plants; and the monitoring of CO and O_2 on fluid catalytic cracking units for safety and catalyst regeneration.

MEECO

FIGURE 3. The NastyBoy online analyzer for measuring moisture in Cl_2 uses materials that resist Cl_2 gas and a design that isolates the electronics from the flow system

Mettler-Toledo brings to the table its GPro 500 TDL series (Figure 2), which offers interference-free, drift-free TDL technology. What makes Mettler Toledo's TDL instruments different is the folded optical path principle. "One drawback of many TDL analyzers on the market that employ a separate laser source and a separate detector is the alignment," he explains. The laser is a very focused stream, but in some across-the-stack installations, it is possible to lose alignment. For example, if the pipe is very large in diameter, it is subject to vibrations, or one side has more thermal expansion than the other, he says. To solve this problem, "we developed a probe design with a folded optical path. This means the laser source and detector are placed in one head that contains all the electronics and is mounted on one side of the pipe or vessel and a probe goes inside the process gas stream containing the reflecting element in the tip. We fold the beam over and back to the analyzer so it's similar to having a mirror in the process that reflects back into the detector. Simply put, it provides the effect of a solid object that doesn't require alignment."

Union Instruments

FIGURE 4. The INCA is a modular device platform for multi-component gas analysis with a particular focus in the biogas and natural-gas industries

Users of devices that measure moisture in Cl_2 are also looking for reduced maintenance requirements associated with calibration and the harsh environment, says Rob Leiter, technical sales representative with MEECO, Inc. (Warrington, Pa.; www.meeco.com). "The real challenge in this application comes from the environment that can corrode almost any metal, including stainless steel," he says. His company offers the NastyBoy online analyzer for measuring moisture in Cl_2 (Figure 3), which provides durability through the use of materials that resist Cl_2 gas and the moisture in Cl_2 gas, and via a design that isolates the electronics from the flow system. Combined, these features help reduce the total cost of ownership and result in less maintenance.

Specifically, the analyzer technology is based on Faraday's law of electrolysis, so the cell absorbs and electrolyzes moisture at fractional parts-per-million or other units of measure. All of the sample moisture (100%) is absorbed by a phosphorus pentoxide film, which covers two spirally wound electrodes that are embedded in a hollow glass tube. When the sample gas enters the cell at a known flowrate, the film absorbs all the water molecules present. By applying an electrical potential (voltage) to the electrodes, each absorbed water molecule is electrolyzed, generating a finite current. This current is precise and proportional to the

Baseline, a MOCON company

FIGURE 5. The Series 9100 gas chromatograph for single- and multi-gas monitoring contains integrated software, eliminating the need for an additional computer

amount of absorbed water. It is, therefore, an exact, direct measurement of the water vapor present in the sample gas.

An added bonus of this technology, according to Leiter, is that the electrolytic cells used do not require calibration in this application, which greatly reduces labor time and costs associated with calibration.

"Any time you can eliminate sample extraction, sample transport and sample systems, you reduce time spent on analysis and eliminate failure points in the overall analysis system."

Jesse Underwood, Yokogawa

Modular systems

"In the chemical industry there are many combustible gases that occur as byproducts of production processes," says Torsten Haug, managing director of Union Instruments GmbH (Karlsruhe, Germany; www.union-instruments.com). "The traditional way to deal with this is flaring the gas with a flare stack. The future will likely see us using these gases for energy production in combustion processes. Therefore, it is important to know the gas properties which are influencing the combustion processes."

For this reason, he says, the market needs a variety of instruments that offer high reliability and easy maintenance, such as automatic calibration and validation functions. Instruments such as BTU analyzers or online measurement of the Wobbe index, heating value, air demand and gas density for all fuel gases and flare gases, will be needed, as well as a variety of sensors with different measuring ranges. Non-dispersive infrared (NDIR) sensors, electrochemical sensors, paramagnetic sensors and acoustic cells will all be needed, and sometimes several may be needed for a single application, he says.

In response, his company offers INCA (Figure 4), which is a modular device platform for multi-component gas analysis with a particular focus in the biogas and natural gas industries. It is similar to modular construction systems, with blocks for active gas sampling and gas processing, as well as sensors, controllers, data processing and communication combined to form reliable analysis systems.

Baseline, a MOCON company

FIGURE 6. The fully automated Series 9000 Hydrocarbon Analyzer offers an automatic calibration feature that enhances long-term stability

"The standardized modules enable a variety of customized solutions for specific analysis tasks and combine optimal analysis results for less cost," Haug says. "The user benefits from the convenience of the modular system, in which individual components can be replaced directly in the field. When equipped accordingly, INCA enables sequential switchover of the measuring technology to different sample gas streams from up to ten measuring points, for example such as sampling from multiple fermenters in biogas plants."

Automation

Analyzers that are smaller and smarter are becoming increasingly sought after, as well, says Hank Braly, vice president of sales and marketing with Baseline, a MOCON company (Lyons, Colo.; www.baseline.com). "Processors want an instrument that can take care of itself via automated calibration against known standards, diagnostics and the ability to connect remotely over the Internet so that we can remotely diagnose, service and make adjustments to instruments located in the field," he says.

At the same time, he adds, reducing the size of the devices is also a goal. "A lot of these instruments are being incorporated into systems with other components, so we are reducing the size of our analyzers. Both the smaller size and automated functions are being made possible due to the increase in processing capabilities of microprocessors that allow us to provide more continuous diagnostics and more sophisticated ways of handling the data."

In addition to offering automatic and remote calibration, Baseline's Series 9100 gas chromatograph for single- and multi-gas monitoring (Figure 5), contains integrated software, eliminating the need for an additional computer. Data collection is in universal text format with user-definable data export utilities for reports and chromatograms.

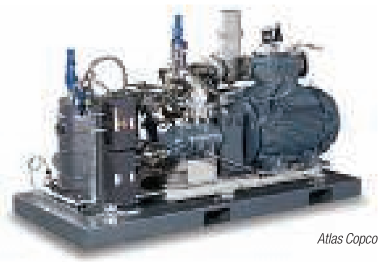
Similarly, the company's fully automated Series 9000 Hydrocarbon Analyzer (Figure 6) also offers an automatic calibration feature that enhances the long-term analytical stability of the instrument. Automatic flame-ionization detector (FID) ignition, automatic shutoff sample, fuel and combustion air, remote operation via RS-232 and Ethernet, and software that allows for remote monitoring and control are also featured.

Whether your gas analysis need is for reduced maintenance, modular instrumentation or automated systems, or any combination of these characteristics, it is likely that analyzer manufacturers are developing a measurement device that will meet your requirements to reduce the labor and other plant resources that were once associated with taking gas measurements. ■

Joy LePree

Compressors, Fans and Blowers


Ametek Rotron


Atlas Copco


Chromalox

This cooling fan is rated for use in explosive environments

The Ametek Rotron MIL-901XP fan (photo) will not create an ignition source in potentially explosive environments, so it is suitable for use in both indoor and outdoor applications where potentially explosive or flammable gases, vapor or dusts may be present, says the manufacturer. Specificially, it meets UL, NEC and ANSI hazardous-use standards. This fan has an ambient operating temperature range of –54 to 70°C. And with permanently lubricated, steel ball bearings, it has a rated life in excess of 200,000 h, ensuring reliability during continuous operations. — *Ametek Rotron, Woodstock, N.Y.*
www.rotron.com

Extreme-duty model extends this family of blower heaters

The CXH-XD blower heater (photo) is designed to withstand corrosive and wet environments and is ATEX-certified. It extends the capabilities of the company's CXH-A units, by providing either primary or supplementary heating (for comfort or freeze protection) in areas that are classified as hazardous locations. CXH-XD enhancements include a corrosion-resistant, coated heat exchanger, optional 316 stainless-steel and epoxy-coated frames, as well as a corrosion-resistant and washdown-rated fan motor. With these added capabilities, this unit is suitable for use in onshore and offshore drilling platforms, pump houses, power-generating stations, water and wastewater plants, chemical storage facilities, pipeline-metering stations and more, says the company. The use of heavy-duty magnetic contactors and a low-voltage transformer simplifies installation. The CXH-XD model comes with power ratings from 3 to 35 kW, which provides heat outputs to 119,420 Btu/h. It is offered for three-phase voltages starting at 240 V, while an optional 690-V design is also available. — *Chromalox, Pittsburgh, Pa.*
www.chromalox.com

These compact gas compressors handle methane

This company's GG-VSD lubricated gas screw compressors (photo) are designed for dry biomethane and methane applications requiring pressure to 16 bars (232 psia). These single-stage compressors are both water cooled and directly driven. The use of a variable-speed drive and a highly efficient rotor profile allows these compressors to maximize flow while minimizing horsepower requirements, and this helps to regulate capacity and keep the inlet pressure constant during operation. The single-stage compressor design is frame-mounted and compact, making it easy to install with minimal floor-space requirements. The GG-VSD compressors comply with ATEX Zone 1 2 design requirements, says the manufacturer.— *Atlas Copco North America, Pine Brook, N.J.*
www.atlascopco.com

Compress low-molecular-weight gases to high pressures

This company's single-shaft, vertical-split-casing turbocompressors are designed to handle a wide range of process gases for both onshore and offshore oil-and-gas applications that require ultra-high pressure processing, including the production of liquefied natural gas (LNG). The STC-SV compressors can handle volume flows from 150 to 280,000 ft^3/min (250 to 480,000 m^3/h), and produce a discharge pressure up to 1,000 bars (14,500 psi), making them ideal for applications involving hydrocracking, synthesis gas or gas storage and injec-

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

tion. These machines are designed according to API 617. The use of a barrel-type design allows for full maintenance without disconnecting process gas lines, says the company. Depending on the application, these turbocompressors can be driven by a steam turbine, gas turbine or electric motor. A smaller unit, called the STC-SV (S), is available as a single-stage machine. With the compressor, driver and auxiliaries constructed using a packaged-unit concept, this turbocompressor eases transport and reduces site installation work, realizing pressures up to 80 bars.— *Siemens AG, Erlangen, Germany.*
www.siemens.com

Explosion-proof fan has rated air delivery of 4,450 ft^3/min

Larson Electronics

The EPF-E16-4450-220V explosion-proof fan and blower (photo) has a Class 1 Division 1 rating and is designed for use on a 220-V a.c. electrical power system. It meets UL and ATEX standards for hazardous locations, says the company. This portable blower features a high-efficiency, 1.5-hp motor, which is fully encapsulated with a precision-balanced, non-sparking aluminum blower blade to both ensure high output and provide protection against accidental ignition. The fan has a 16-in. intake and a 15-ft static duct to direct stale air from hazardous location areas. It produces 4,450 ft^3/min of air output for robust airflow in larger spaces. Ambient operating temperature for this blower ranges from –20 to 180°F.— *Larson Electronics, Kemp, Tex.*
www.larsonelectronics.com

Positive-displacement blowers move process and cooling gas

The Series GQ process gas blowers (photo) can be used to convey a wide array of process or cooling gases and gas mixtures, across a range of industrial applications. Six different models are available to handle volume flow ranges from 1,500 to 100,000 m^3/h, at operating pressure ranging from 2.5 to 6.0 bars. The conveying chamber uses single-acting, oil-purged mechanical seals in combination with labyrinth seals. The drive shaft is sealed by three radial seal rings. The direction of flow is horizontal and can be directed toward the left or to the right. The drive relies on a flexibly coupled spur gear or direct coupling with a drive motor, or via a flexibly coupled spur gear. — *Aerzen USA Corp., Coatesville, Pa.*
www.aerzenusa.com

Aerzen

Circle 7 on p. 78 or go to adlinks.chemengonline.com/56196-07

Reciprocating air compressor has built-in backup capacity

The Duplex reciprocating air compressors feature two individual compressor pumps and two motors mounted on a single tank, ensuring 100% built-in backup or additional air power for larger-capacity applications. The Duplex is ideal for applications where available power does not allow for a single larger compressor, says the company. Its relatively compact size also makes it ideal for use in tight spaces. Manufactured from durable cast iron, the unit is designed to provide automatic duplexing of or alternating of compressors, depending on compressed air reqruiements. It provides 175-psig maximum operating pressure for applications that require high air capacity and pressure. It also features an ASME-coded receiver tank, a mounted and wired magnetic starter and alternator panel, and automatic start/stop controls. Optional vibration isolators are available.— *Ingersoll Rand, Davidson, N.C.*
www.ingersollrand.com

Rugged air compressors are built to move


Kaeser Compressors

The Mobilair Series portable air compressors (photo) is available to provide a source of compressed air for onsite use. These units provide capacities from 50 to 1,200 ft^3/min, in pressures from 100 to 145 psig (with selected models available to provide pressure to 205 psig). The diesel-driven air compressors have a large-capacity fuel tank, which provides extended operation — more than 10 h of run time — and has a large filler neck with overflow protection for safe, easy filling. These mobile units are constructed from durable, industrial-grade materials that resist rust and corrosion, and they are designed to minimize noise. Towable compressors feature torsion bar suspension, over-sized tires and a height-adjustable tow bar for reliable road handling. — *Kaeser Compressors, Fredericksburg, Va.*
www.kaeser.com

Sealless compressors avoid process gas contamination


Sundyne

This company's PPI Diaphragm Compressors are hermetically sealed, positive-displacement machines that completely isolate lubricants from the process gas. This sealless design (which is devoid of any dynamic seals) are designed for applications that require leak-free, contamination-free operation. One specific unit, the Sundyne Series 7L Diaphragm Compressor (photo), is available in single- and two-stage configurations. This compressor has a maximum working pressure of 6,250 psi, with a maximum motor size of 200 hp and 149 kW.With a maximum displacement of 110.9 ft^3/min, the compressor has a maximum speed range of 250 to 350 rpm, and a maximum discharge temperature of 450°F (232°C). All Sundyne PPI diaphragm compressors can be custom packaged for specific applictions requiring API-618 standards.— *Sundyne, Arvada, Colo.*
www.syndyne.com

Rotary positive blowers can be custom engineered

This company's M-D Pneumatics rotary positive blowers and blower packages are designed for use in wastewater-treatment facilities and other industrial applications. The treatment of wastewater requires large volumes of oxygen to be delivered to aeration tanks or ponds, to support the growth of microorganisms that consume organic matter. Such facilities also require blowers to support

the transfer of digester gases that are produced. A variety of standard blowers and blower packages are available, and designs can be custom engineered to meet the facility's site-specific requirements by customizing the blower design, materials of construction, seals and accessories. Multistage blower packages are available for high-pressure operation. The company offers a wide array of testing services, such as ASME PTC-9 testing, hydrostatic testing, pressure-gas testing, seal-leakage testing, noise testing and non-destructive testing of components. — *Tuthill Vacuum & Blower Systems, Springfield, Mo.*
www.tuthill.com

Blowers ensure 100% oil-free air delivery for many uses

A complete range of Tri-Lobe Blowers is available with flowrates from 10–15,000 m^3/h in single stage, and up to any capacity in parallel configurations, for working pressures to 1 kg/cm^2. They can be purchased as standalone blowers for replacement into an existing system, or as a complete packaged unit. These blowers ensure 100% oil-free air delivery. They have no vanes, valves or rings to wear, and their large inlet and outlet connections ensure minimum losses. These blowers are available for use in a broad range of applications, including water treatment (for backwashing of filtration beds), effluent treatment (to provide diffused aeration and agitation of effluent), cement production (for blending aeration, fluidization and conveying), chemical process operations (to supply process air), bag filtration (to support reverse cleaning of filter bags) and more. — *Everest Blowers, New Delhi, India*
www.everestblowers.com

Online selection guide supports blower selection

This company's oil-less vortex blowers are configured for use in a variety of demanding industrial environments. With user-friendly operator interface and compact design, they are designed to reduce ongoingmaintenance requirements. The E-Series High Flow Vortex Blowers are equipped with cup-shaped impellers that generate air via non-interference and single-path air compression, making them ideal for large-air-volume applications. The G-Series High Pressure Vortex Blowers have specially designed impellers that produce high-speed air by maintaining an optimal vane surface, thereby allowing higher pressures to be produced for different discharge and vacuum applications. These blowers can be custom engineered to withstand extreme temperature and pressure conditions, to ensure long operating life and minimal maintenance. The company offers online tools to help engineers match the blower to the application.— *Hitachi America, Ltd., Tarrytown, N.Y.*
www.hitachi-america.us ■

Suzanne Shelley


HPC Compressed Air Systems


WIKA Alexander Wiegand SE & Co.


Automatik Pelletizing Systems


Microdyn-Nadir

Integrated blowers save time and money

These new compact rotary blowers (photo) are supplied connection-ready with integrated mains power supply equipment and monitoring, including all sensors and star-delta starter, or with a frequency converter to enable flexible speed control. The range BB69C through FB791C rotary blowers deliver from 0.5 to 74 m^3/min at up to 1,000 mbarg, or 500 mbar (partial vacuum), and are suitable for applications such as pneumatic conveying of powdered goods, water treatment, or wherever the generation of blowing air is required from a compact and efficient unit. The complete system packages are CE- and EMC-certified, which means less work for both the operator and system provider in terms of planning, installation, certification, documentation and commissioning. — *HPC Compressed Air Systems, Burgess Hill, U.K.*
www.hpccompressors.co.uk

A new level sensor in a hygienic design

For sanitary applications, this manufacturer has developed a magnetostrictive level sensor in hygienic design. The model FLM-H sensor (photo), is certified in accordance with 3A Sanitary Standards, and measures level in tanks and vessels with an accuracy of less than 0.5 mm. The level is detected continuously, regardless of any physicochemical changes of state in the media, such as foaming or boiling effects. Based on the float principle, the device even operates reliably with clean- and steam-in-place (CIP/SIP) processes. The 4–20-mA output of the sensor (also available with HART protocol) transmits the signal over long distances. The sensor is also suitable for SIL 2 (IEC 61508) installations. — *WIKA Alexander Wiegand SE & Co. KG, Klingenberg, Germany*
www.wika.com

A mobile app for catalyst recommendations

This company has launched its Catalytic Reaction Guide (CRG) as a free mobile application (app), which provides users mobile access to catalyst recommendations on over 150 chemical reactions in the area of homogeneous, heterogeneous and asymmetric catalysis. Users can easily view and search a full product listing with streamlined technical support, take advantage of an efficient quotation request process, and obtain contact information for a complete services offering. In addition to catalyst recommendations, the app features a convenient *Chemical Abstracts Service* searchable product listing and relevant literature references. The interface is fully customizable through a user profile that synchronizes data with other devices for easy tracking of recommendations and details. — *Johnson Matthey Catalysis and Chiral Technologies, West Deptford, N.J.*
www.jmcct.com/crg

This pelletizer incorporates a number of features

The Primo 200E (photo) is said to be particularly well-suited for the compounding of thermoplastic masterbatches with throughputs of up to 1.5 ton/h. The Primo 200E is a single-side mounted, dry-cut strand pelletizer with an extra-large cutting width of 200 mm. Its unique cutting geometry — with the shortest, unguided length between the feed rollers and cut — permits optimal straight cutting of both hard, abrasive and very soft, flexible plastic strands. The pellet dimensions can be changed very quickly thanks to an optional automatic pellet-length adjustment system. — *Automatik Pelletizing Systems, a subsidiary of Maag — a Dover Corporation company, Oberglatt, Switzerland*
www.maag.com

These membrane modules now boast a larger area

This manufacturer has increased the membrane area of its Bio-Cel BC400 by 4%, and thus changes the name to BC416 (photo). The increase in membrane area allows for an optimization of the crossflow, as well as enabling an increased packing density. These changes will also be implemented with the BC100 (soon to be called Bio-Cel BC104) modules throughout the course of 2015. — *Microdyn-Nadir GmbH, Wiesbaden, Germany*
www.microdyn-nadir.de

A new silicone resin for water-repellent wood coatings

Presented at the European Coatings Show (Nuremberg, Germany; April 21–23), the new silicone-resin emulsion Silres WH is based on a new silicone resin, and can be used as a hydrophobizing wood-impregnating agent or as an additive in wood stains. Wood impregnated with Silres WH is protected against dampness, moisture and the subsequent damage caused by fungi and insects for an exceedingly long time. The company is also showcasing a binder from its new Primus line for high-value outdoor applications. Primis AF 1000, the line's first product, combines organic and inorganic components, enabling the formulation of bright, highly durable exterior paints with reduced dirt pick-up. — *Wacker Chemie AG, Munich, Germany*

www.wacker.com

This glass flowmeter now has a 4–20-mA output option

With the new option "WIM", a 4–20-mA electrical output signal is now available for the variable-area flowmeter VA40 (photo). While the flowrate with these glass devices could previously be read only locally, the 4–20-mA output option allows for transmission of the measured value for control or evaluation purposes. With VA40 WIM version, the height of the float is converted into an electrical signal via a Hall sensor chain. This signal can be used for a PLC/DCS, or to record the measured value over a certain period with a data logger. Along with each VA40 WIM, the manufacturer supplies the calibration curve, allowing the customer to assign the exact flow value to any current value. — *Krohne Messtechnik GmbH, Duisburg, Germany*

www.krohne.com

Gas analysis in natural gas and biogas plants

Inca (photo) is a modular device platform for multicomponent gas analysis with a particular focus in the biogas and natural-gas industries. Similar to a modular construction system, blocks for active-gas sampling, gas processing, as well as sensors, controller, data processing and communication are combined to form robust and reliable analysis systems. The standardized modules enable a variety of customized solutions for specific analysis tasks and combine optimal analysis results with a favorable cost position. When equipped accordingly, Inca enables sequential switchover of the measuring technology to different sample-gas streams from up to ten measuring points, for example, for sampling from multiple fermenters in biogas plants. A variety of sensors with different measuring ranges are available for the devices, including NDIR sensors (for CH_4 and CO_2), electrochemical sensors (for O_2, H_2S and H_2), paramagnetic sensors (for O_2) and acoustic cells (for specific density). — *Union Instruments GmbH, Karlsruhe, Germany*

www.union-instruments.com

Datasheet-management platform with GHS-specific enhancements

Online-SDS 9.0 is the newest available version of this company's application for managing chemical and safety datasheets (SDS). This latest edition features enhancements designed to improve the accessibility and usefulness of compliance information stored within the platform. With hazard classification information changing rapidly across the world due to the Globally Harmonized System (GHS) for the Classification and Labelling of Chemicals, this online application provides easily accessible navigation and tracking of metrics, such as volume of SDS per location and the volume of new products added or removed per month. Other improvements include new export options for product data and streamlined report pages. — *3E Co., Carlsbad, Calif.*

www.3ecompany.com

Isolate dust explosions with this updated line of equipment

The Exkop combutible-dust-explosion isolation system (photo) provides safe and economic explosion, spark and flame-front isolation. The Exkop system is now available for an expanded range of applications, including those with reduced explosion pressures and larger diameters than were previously served. The pinch valves protect interconnected equipment via the high-speed closure of a fast-acting food-grade elastomer hose. — *Rembe, Inc., Charlotte, N.C.*

www.rembe.us

Krohne Messtechnik

Union Instruments

Rembe


Seepex

Charles Ross & Son Co.

Explosion Power


QED Environmental Systems


Intertec Instrumentation

A programmable module enables pre-configured pump parameters

The Intelligent Metering Pump (photo) incorporates an Electronic Programming Module (EPM) memory chip on which parameters can be pre-configured. This makes replacement of a drive simple, quick and accurate. Users can simply insert the EPM chip from a previous drive into the new one with no downtime. When the EPM-equipped drive is used on a line containing multiple drives with identical setup, programming the entire line takes just minutes. Various accessories can easily be connected to the drive via the control terminals on the terminal strip inside the controller. A control module is also available as an option, but it is not needed when accessories are wired directly to the terminal strip. — *Seepex Inc., Enon, Ohio*
www.seepex.com

This disperser re-suspends densely packed, layered materials

This company's bowtie dispersers (photo) are designed for mixing heavy pastes and viscous liquids, with handling capability of viscosities up to several hundred thousand centipoise. Driven by an explosion-proof inverter-duty motor, the bowtie agitator creates a very uniform consistency without running at high speeds. This type of mixer is also suitable for re-suspending dense materials that have packed into layers during storage or transit. An air-oil hydraulic lift raises and lowers the agitator into the vessel. Safety limit switches prevent operation of the mixer while in the raised position or without a vessel in place. All wetted parts are 304 stainless steel. — *Charles Ross & Son Co., Hauppauge, N.Y.*
www.mixers.com

Online boiler cleaning with this shock-pulse machine

The Shock Pulse Generator (photo) automatically generates shock pulses, providing continuous boiler cleaning in a number of applications, including waste-to-energy plants, industrial boilers, sludge-incineration plants, coal-fired power plants, black liquor boilers, filters, spray absorbers and cement kilns. The small and compact design makes it easy to install in existing and new plants. The Shock Pulse Generator's online boiler-cleaning technology reduces standstill periods, increasing overall boiler efficiency. — *Explosion Power GmbH, Lenzburg, Switzerland*
www.explosionpower.ch

A leak-tight barb fitting for handling liquids and gases

The patent-pending Easy Port Capped Barb Fitting (photo) is a sampling port designed for liquids and gases under vacuum conditions or in low-pressure applications. Unlike standard barb fittings, the Easy Port includes a leak-tight, screw-on cap to seal the barb between uses. The cap also protects the barb from contamination and damage, and is tethered, so it cannot be dropped or lost. Made with tough, fiber-reinforced nylon for high strength, these barb fittings are also ultraviolet (UV) protected. The fittings' heavy walls help to reduce heat loss and freezing. — *QED Environmental Systems, Inc., Ann Arbor, Mich.*
www.qedenv.com

A large-capacity sunshade for shielding multiple instruments

The CubeShade (photo) is a large, cube-shaped sunshade for process instrumentation that shields equipment, such as electronic monitoring systems, explosion-proof junction boxes or analyzer installations, from solar radiation. The CubeShade's protective cover measures 600 × 550 × 500 mm, providing a large shaded environment that can accommodate and protect large or multiple instruments, as well as simplifying maintenance access. The CubeShade is constructed entirely from glass-fiber reinforced, sheet-molding compound (SMC). The material's advantages include a high resistance to UV light and corrosion from salt and common petrochemicals, as well as a low thermal conductivity, which helps to prevent the transfer of heat-generating solar radiation into the shaded area. — *Intertec Instrumentation Ltd., Sarnia, Ont., Canada*
www.intertec-inst.com ■

Gerald Ondrey and Mary Page Bailey

Industrial Microorganisms

Department Editor: Scott Jenkins

Evidence for human use of microorganisms in the production of valuable goods extends back thousands of years. Over the ensuing centuries, the use of microbes in industrial processes has grown steadily, and continues today. This one-page reference provides information on the diversity of industrial processes that utilize microbes, including examples of microbe-dependent products.

Metabolic products

Traditionally, microbes used in fermentation processes have been naturally occurring strains of bacteria and molds that carry out a specific metabolic reaction on a substrate. In the last 25 years, industrial microbes have increasingly been mutant strains engineered to selectively synthesize maximized amounts of various metabolic intermediates. The products of an industrial microbial process can be divided into two broad classes: primary metabolites (produced within the microbes' major metabolic pathways and essential for microbes' function), and secondary metabolites (byproducts of metabolism that may not be critical to the microbes' function).

Desirable properties

Desirable qualities for effective microorganisms in industrial use include the following:

- Ability to grow in culture
- Genetic stability
- Ability to efficiently produce a target product in a short time period
- Limited need for additional growth factors
- Utilization of a wide range of low-cost and readily available carbon sources
- Amenability to genetic engineering
- Non-pathogenicity
- Readily harvested from the fermentation process
- Limited production of byproducts to simplify purification
- Production of spores or other reproductive cells (to allow inoculation into large fermenters)
- Ability to protect itself from contamination by other microbes

TABLE 1. SELECTED INDUSTRIAL APPLICATIONS THAT DEPEND ON MICROORGANISMS AND CORRESPONDING PRODUCTS

Application	Example products or processes
Food and beverage production	Yeast is used in beer brewing and wine making; Fungi and bacteria are used in the manufacture of dairy foods, including cheese, yogurt, sour cream, cottage cheese and others; Various microbes are use in making pickled products, soy sauce, sauerkraut, kim chee and other fermented foods
Wastewater treatment	Industrial and municipal wastewater can be treated with mixed microbial populations to remove suspended solids, dissolved organic compounds and to oxidize ammonia
Bioremediation	Naturally occurring bacteria, fungi and algae have been used to remove pollutants, such as polycyclic aromatic hydrocarbons, halogenated hydrocarbons, heavy metals and others, from soil
Biocatalysis	Microbes are used to produce specific enzymes, such as lipases, amylases, DNA polymerases, serine proteases and others, which are then used to catalyze other reactions
Biofuel production	Bacteria, yeast and algae have been used to manufacture bioethanol and biodiesel as transportation fuels, as well as biomethane and bio-derived hydrogen
Bio-based chemical production	Engineered bacteria and algae have been used to manufacture organic intermediate chemicals and solvents, such as acetone, bio-butanol, ethanol, lactic acid, succinic acid, butanediol, propanediol, butadiene and others
Pharmaceutical production	Bacteria and fungi have been used to produce several classes of antibiotics, antifungals, chemotherapeutic agents, enzymes, proteins, nucleic acids and vaccines
Biopolymers	A number of polymers, such as polyhydroxyalkanoates (PHA), polyesters, polysaccharides and polyamides are synthesized with the help of microorganisms
Bioleaching	Bacteria have been used to extract metal ions (including gold, copper and cobalt) from ores, especially low-grade ores, as well as from sludges and wastes
Biobleaching	Microbes have been used to remove lignins from wood pulp in the paper industry
Food additives	Bacteria and fungi have been used to manufacture a wide range of food additives, such as polyhydric alcohols (erythritol, mannitol, and so on), citric acid, xanthan gum, vitamin C and other vitamins, as well as flavors and colors
Genetic enginering	Viruses have been used as delivery vectors to insert genetic material into other cells

- Relatively large cell size, to allow faster settling or simpler filtering

Microbe types

Industrial microorganisms generally fall into one of the following categories: yeasts, fungi (other than yeast), algae, bacteria, archaea and viruses.

Algae. The term algae refers to an extremely diverse group of eukaryotes (organisms with defined cellular nuclei), strains of which are used industrially in the production of biofuels, such as biodiesel, and in wastewater treatment, among other uses.

Bacteria. Among the earliest life forms, bacteria are prokaryotes (lacking defined nuclei) with staggering metabolic diversity. Industrially, many different bacterial species are used in a wide range of processes from biofuels to pharmaceuticals.

Archaea. Similar in many ways to bacteria, archaea constitute a separate class of organisms with distinct metabolic pathways and unique biochemistry. Archaea find limited industrial use, but are used in biogas production, sewage treatment and as a source of heat-tolerant enzymes.

Fungi. A family of eukaryotes that includes yeasts, molds and mushrooms, fungi have long been used to produce antibiotics in industry.

Yeast. Likely the oldest domesticated microorganism, yeast is a single-celled eukaryote best known industrially for producing ethanol from sugars by fermentation.

Viruses. Viruses can be used as delivery vectors for introducing genetic material into other cells.

Key industrial microbe species

- *Saccharomyces cerevisiae* (yeast for brewing and bread-making)
- *Escherichia coli* (bacteria for recombinant proteins and others)
- *Aspergillus niger* (fungus for manufacturing citric acid and enzymes)
- *Clostridium butyclicum* (bacteria used in soured milk and cheeses)
- *Xanthomonas campestri* (bacteria that produces xanthan gum)
- *Deinococcus radiorans* (bacteria for soil and water remediation) ■

For a list of reference material, view the online version of this column at www.chemengonline.com

Onsite Enzyme Production

By Intratec Solutions

Enzymes are proteins synthesized in living cells that act as biocatalysts for specific reactions. However, enzymes must be applied under mild conditions, since they lose their catalytic activity when subjected to heat, organic solvents or strong acids and bases.

Enzymes are industrially produced from the fermentation of microorganisms, but they can also be obtained from plant and animal sources. They offer high specificity and selectivity for reactions and substrates, and can produce enantiopure compounds (in contrast to chemical syntheses, which usually produce racemic mixtures).

Enzymes are applied in the production of food, fine chemicals and pharmaceuticals. In addition, they are used in washing processes, as well as for a wide range of analytical purposes. Alternatively, enzyme technology has been developed to improve sustainable processes that produce existing products using novel raw materials, such as biomass.

The process

A typical process for onsite enzyme production through fermentation is described below, using the enzyme cellulase as an example. Cellulase is used in the hydrolysis of lignocellulosic material in biomass-based processes, such as the production of cellulosic ethanol. Cellulase is industrially produced by a fungus in an aerobic fermentation in the presence of a cellulase-production inducer (Figure 1). The process shown was compiled from a technical report published by the National Renewable Energy Laboratory (NREL; Golden, Colo.; www.nrel.gov).

Medium preparation. Glucose feedstock is mixed with water and a small quantity of cellulase, which converts part of the glucose to sophorose, a cellulase-production inducer.

Pre-fermentation. The fermentation inoculum is prepared in a batch pre-fermentation step, which occurs in four trains with three pre-fermenters each, into which part of the glucose medium, nutrients and air are fed.

Fermentation. The fermentation step is conducted in nine fermenters, which are fed with the fermentation inoculum, culture medium, nutrients and air. The fermentation is a fed-batch process, in which, after the depletion of nutrients caused by cell growth, glucose medium is fed to the reactors, allowing continuous enzyme production. After fermentation, the cellulase-containing broth can be directly fed to the hydrolysis section of a cellulosic ethanol facility, since the high temperature applied in this operation is sufficient to inactivate the microorganism.

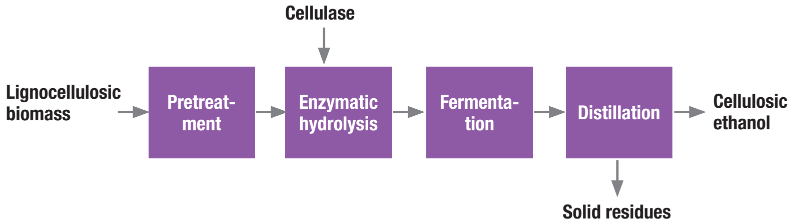


FIGURE 2. A typical cellulosic ethanol production process is shown here

Economic evaluation

An economic evaluation of the process was conducted based on data from Q2 2014. The following assumptions were considered:

- A cellulase production unit constructed in the same complex as a cellulosic ethanol facility located on the U.S. Gulf Coast
- Onsite enzyme production unit manufactures 5,200 ton/yr of cellulase
- Storage and utility facilities were not considered

Total fixed investment for the construction of this unit was estimated at about $40 million.

Global perspective

Cellulase enzyme is applied to hydrolyze lignocellulosic biomass, which is used as feedstock in the production of cellulosic ethanol (Figure 2). In this way, cellulosic ethanol costs are highly related to enzyme costs.

Initially, enzyme costs were too high, so cellulosic ethanol was not cost-competitive with corn-based ethanol. Currently, many advances in enzyme technology have been achieved, lowering enzyme costs in cellulosic ethanol by both enzyme production-cost reduction and enzyme efficiency enhancement. In addition, onsite production allows the manufacture of the desired enzyme at lower costs, since some downstream operations present in a standalone enzyme unit (such as purification, formulation and storage), as well as transportation of the enzyme product, are not necessary. ■

For more on industrial enzymes, see "Tunable Enzymes and the Leaner, Greener CPI" in the January 2015 issue of *Chem. Eng.* at www.chemengonline.com

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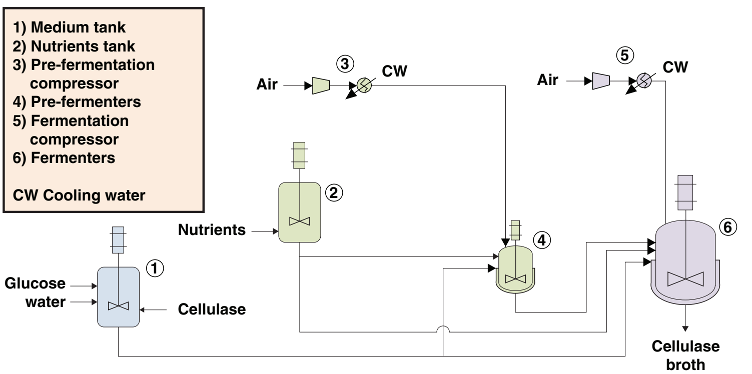


FIGURE 1. The process diagram shows onsite cellulase enzyme production

Column Revamps: From Outside to Inside

Some of the many things to consider for this complex task are presented here

Mike Resetarits
Separations Consultant
Tim ReBeau
Gallop Field Services
Terry Thurber
Fractionation Research Inc.

IN BRIEF

FIGURE 1. Revamping a column is a major undertaking

Petroleum refineries, chemical-processing plants, natural-gas purification plants and other facilities in the chemical process industries (CPI) all contain columns. These columns perform distillations [*1*], regenerations, strippings and absorptions. Column diameters range from 12 in. to 50 ft, and column heights range from 10 to 350 ft.

Prior to about 1970, the majority of the column work performed in the U.S. was the building and installation of new, grass roots, columns. Subsequent to that, the workload shifted, with many existing columns being revamped to achieve performance improvements or maintenance targets. When columns are revamped, some columns are revamped on the outside; some, on the inside (Figure 1); and some, both.

Associated with columns are heat exchangers, reboilers, condensers, receivers, pumps, valves, tanks and instrumentation. Most column revamps necessarily include evaluations and modifications of this ancillary equipment.

Unfortunately for us, no two real column revamps are identical. They are never easy. And column revamps cannot be boiled down to a step-by-step procedure that can be applied to most columns. Instead, this article presents some of the many considerations that are involved in column revamps.

Trays and packings

Today, globally, approximately one half of columns are equipped with trays and the other half contain packings [*2*]. The packings can be random or structured. For many

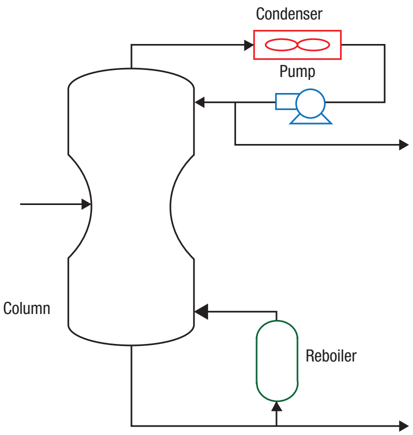


FIGURE 2. Sometimes the column bottlenecks the unit

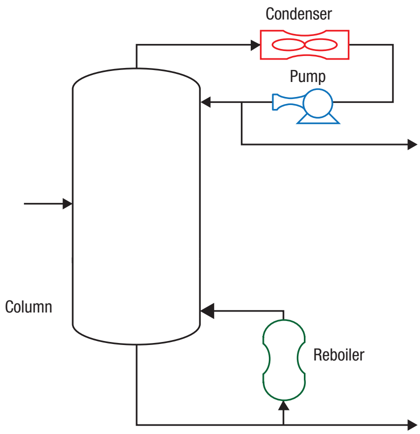


FIGURE 3. Sometimes the ancillary equipment bottlenecks the unit

years, the performance of trays was considered to be more predictable than packing. Around 1980, however, work by Fractionation Research Inc. (FRI; Stillwater, Okla.; www.fri.org), and others, showed that successful packing performance usually hinges on proper liquid distribution above packed beds. At about that same time, sheet-metal structured packing was invented, and such packing sometimes showed advantages over both random packings and trays.

When compared to trays, the biggest advantage attributable to packings is lower pressure drops. This is particularly true with columns that are operating at pressures below atmospheric. As an example, prior to about 1985, most ethylbenzene-styrene vacuum columns were trayed. Many of those columns were soon revamped with structured packings to reduce the pressure drops across the columns, to reduce the bottom temperatures and to reduce renegade polymerization. Generally, the pressure drop across a bed of structured packing is about one-sixth of that across a stack of trays having the same total height.

On the other hand, there are certainly some services where packing seems to be inapplicable. High-pressure distillation columns, where pure products are being targeted, have resisted the packing "revolution." Trays perform better, and much more predictably, in such columns where the volumetric liquid-to-vapor ratios are high. Regarding such packed columns, some distillation theoreticians contend that the high liquid rates are causing vapor to back-mix downward. Some theoreticians contend that the low surface tensions of the liquid phases cause tiny liquid droplets to be sheared off easily from the downward-flowing liquid films; those droplets are entrained detrimentally upwards in such columns.

Random packings (for instance, Pall rings) are still very viable in many columns. Random packings are easy to install and have low pressure drops. Their optimum performance depends upon the liquid distributors that are feeding the packed beds. Good liquid distribution is also, of course, required with structured packings.

Regardless of the tower internals, fouling is never good. With trays, some parts get plugged up and are no longer capable of passing liquid or vapor. With packings, distributors become plugged and liquid distributions become non-optimum. Structured packings are much more prone to fouling than random packings, because the metal sheets of structured packings are very tightly spaced.

The companies that develop and sell trays and packings have been very successful at improving the performances of those mass-transfer products. Subsequent to about 1960, there have been four generations of trays as follows:

- Crossflow
- Augmented crossflow
- Counterflow
- Cocurrent flow

Generally, each generation had a higher capacity. Some generations exhibit higher efficiencies than others.

Some practitioners divide the history of random packings into four generations. First came Raschig rings and Berl saddles; then, Pall rings and Intalox saddles; then, proprietary rings from several vendors; and then, high-performance rings from several vendors.

Structured packings had the following history:

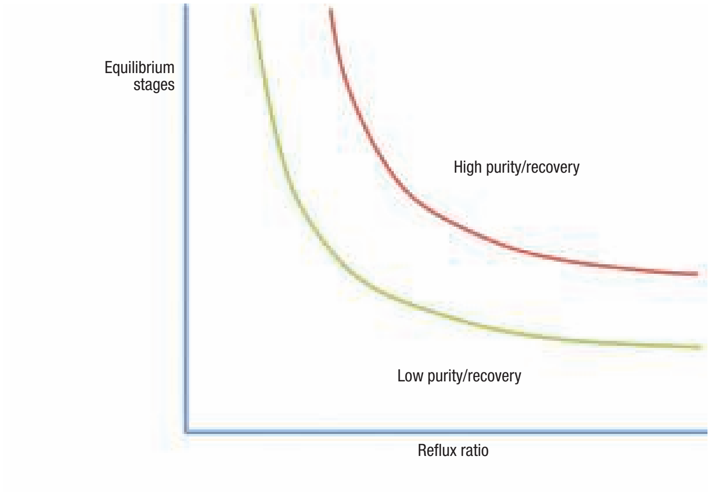


FIGURE 4. Attainment of purity and recovery target depend upon the number of equilibrium stages and the reflux ratio

- Gauze
- Corrugated and textured sheet metal
- High-capacity corrugated and textured sheet metal

High-capacity structured packings generally exhibit about 20% more capacity than "conventional" sheet-metal structured packings, with no loss in efficiency.

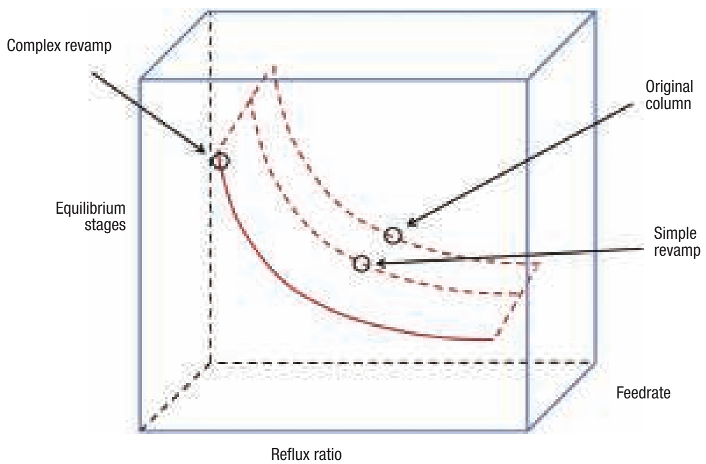
All of these new generations of trays, random packings and structured packings have made it easily possible, at times, to make large improvements in column performances with relatively easy column revamps.

Many column revamps include the changing out of the column internals. These change-outs can be divided into the following broad categories:

1. Trays replace trays
2. Trays replace packing
3. Packing replaces packing
4. Packing replaces trays

When trays replace packings, it is usually because the packing performance has been disappointing. When packing replaces trays, it is usually in pursuit of reduced pressure drops.

FIGURE 5. A simple revamp can yield a feedrate increase and there are times when a complex revamp can yield a larger feedrate increase [*2*]



Revamp targets

Some columns are revamped on the outside. Old insulation is removed. The bare tower is sand-blasted, re-painted and then reinsulated. Such work is required to properly address corrosion under insulation (CUI), which is an increasing safety problem with the aged and aging columns of the western world. After painting, some of these columns are left uninsulated. This allows for easy determinations of future metal-thickness losses almost everywhere on the column shell.

Some columns are revamped on the inside, which is the primary topic of this article. The possible targets of such revamps are the following:

- Replacement, in kind, of old, corroded or plugged, internals
- Increased capacity
- Improved separation (better purities, recoveries or both)
- Reduced energy consumption

In some cases, a column's service (key separation) is changed completely. Such a revamp can prove to be particularly challenging.

A simple replacement of old internals requires that details of those internals are available via drawings or purchase orders (POs). Such drawings and POs often prove to be outdated, and wrong. Whenever possible, before such a revamp, the tower should be entered to ascertain exactly what is presently residing inside the tower.

All of the other revamp targets require the performance of process simulations work. Many such computer programs are available commercially. For distillation columns, "simple" programs employing equilibrium stages, or "theoretical trays," suffice. Carbon-dioxide absorbers and regenerators are a completely different case. Such columns often exhibit and require very few equilibrium stages. For example, a carbon-dioxide absorber might require 0.8, or 3.2, equilibrium stages. For such columns, computer programs that utilize the rate-based (approach to equilibrium) calculation strategy will give more satisfying results.

Some engineers might argue that a revamp targeting a simple capacity increase does not require a process simulation. This is not true. The highest volumetric flowrates inside a tower are rarely determinable from simple mass balances of the feed and product streams. Volumetric flowrate

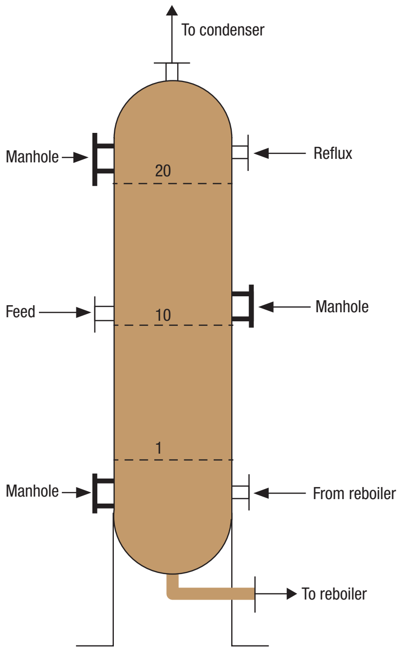


FIGURE 6. Columns contain nozzles for feeds and draws and also manholes for human entry [*2*]

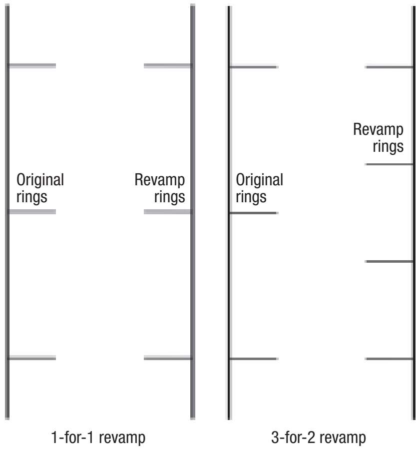


FIGURE 7. Trays can be changed out on a one-for-one basis, or revamps can be more complex

bulges occur inside columns. Also, stage-by-stage densities, viscosities and surface tensions are required. Feed flashes might cause the above-feed tray or packing layer to be the most congested. Old, original, process simulations often prove to be misleading when employed to evaluate current processing conditions.

Computer simulations of columns will also help to identify bottlenecks in the columns' ancillary equipment. Too often, columns are revamped with superior internals, and eventually, it may be determined that the old internals were not bottlenecking the unit; the problem might have been reboiler circuitry piping, an under-sized reflux pump, or condenser cooling water temperature, and so on [*3*]. Figure 2 illustrates a tower that is bottlenecked by its internals (trays or packings). Figure 3 demonstrates that the ancillary equipment often bottlenecks performance.

The target of some column revamps is an improved separation. Maybe a top product needs to be more pure. Maybe the losses of that product, out the bottom of the tower, need to be decreased. The pursuit of such targets absolutely requires the performance of process simulation work. Such work will sometimes include a multiplicity of feed cases, such as near-term, future, worst-case, best-case or other scenario. A serious revamp study usually begins with a simulation of the existing operation. In a perfect world, a perfect vapor-liquid equilibrium (VLE) thermodynamic model is employed in that simulation. A somewhat imperfect model can suffice as long as the number of equilibrium stages being exhibited by the existing, old trays or packings is reasonable. For example, a process simulation showing that 130 theoretical trays are being exhibited by 100 actual trays is not reasonable. The VLE model is probably wrong and a process simulation of the future conditions should not be trusted. For this particular example, a process simulation showing that 90 theoretical trays are being exhibited is reasonable. That VLE model is possibly "good enough."

The target of some column revamps is energy reduction. Figure 4 applies to distillation columns. Two curves show the well-known distillation relationship between the number of equilibrium stages (NES) and the reflux ratio. The *y*-axis of this graph could be relabeled as number of theoretical trays (NTT). The present *x*-axis shows reflux ratio, but this axis could be changed to show energy consumption. Both of the curves of Figure 4 show that as the staging in a column is increased, the energy requirement decreases. There are minima, however, on staging and on reflux ratio. Figure 4 includes two curves, one for an easy separation and one for a difficult separation. As expected, the latter requires more

Source: FRI

FIGURE 8. New tray rings can be welded to tower walls or they can be supported using posts that are attached to the old rings

stages, more energy or both.

Most importantly, Figure 4 shows that energy consumption can be reduced with more staging. More staging can be obtained with more trays, more-efficient trays, more packing or more-efficient packing. One way to place more trays or more packing in a column is to increase the height of the column. Indeed, sometimes, the head is cut off of an old column and a new spool piece is added to the shell to increase the column's height.

Usually, however, increased staging is achieved as follows:

1. Adding trays by decreasing tray spacings, for example, 100 trays at 24-in. spacings are replaced by 150 trays at 16-in. spacings
2. Replacing random packing with more-efficient (that is, smaller) packing elements
3. Replacing structured packing with more-efficient (that is, higher-surface area) structured packing

In all of these cases, the new trays or the new packings must be capable of handling the new internal flowrates, without flooding.

Figure 5 is a three-dimensional graph. The datum labeled "original column" shows the "as is" operating point. A feedrate increase could be achieved with a simple revamp, for example, higher capacity trays or higher capacity packings. Assuming that there were no changes in the tray or packing efficiencies after the simple revamp, the column would be operating at a higher reflux rate, but the same reflux ratio. With a simple revamp, the staging in the column is not changed.

Now imagine a complex revamp — for example, the replacement of 100 trays with 150 trays. Assuming a tray efficiency of 80%, the number of equilibrium stages supplied by the trays would be increased from 80 to 120. This has a large impact on the requisite reflux ratio, as shown in Figures 4 and 5. The reduced reflux ratio leads to a decrease in the reflux rate, a decrease in the reboiler boil-up and decreases in the internal vapor and liquid flowrates. The reduced internal flowrates make it possible to increase the feedrate to the column, as shown in the datum labeled "complex revamp." Similarly, high-efficiency packings could replace less-efficient packings to achieve the benefits of a complex column revamp.

Mechanical considerations

Figure 6 shows a very short distillation column with only 20 trays. Included is a feed nozzle half-way up the column. Process simulation work on any distillation column shows that there is an optimum feed point location, where energy consumption is minimized. That optimum location could be in the upper half or the lower half of the column. During some distillation column revamps, the feed location is changed. The obvious way to move a column's feed point is to add a new nozzle to the column. This is not always easy. Another way to change a feed location is to use the old, original feed nozzle and then run a pipe up or down through a set of trays (or packings) and then terminate that pipe with horizontal distributor piping at the optimum feed location. Primarily, this internal piping strategy avoids welding a new nozzle to the outside of an old column.

An explosion that occurred in 1984 in Lemont, Ill., served as a wake-up call to global column-revamp practitioners. When welding work is performed on "old" pressure vessels, that work must be followed by local or total heat treating (stress relief). Such heat treating is expensive when performed correctly.

In columns, trays are supported by rings. With a new column, those rings are often welded to the inside of the column while the column is in the fabrication shop. Eventually, the column is shipped to the production site and the column is erected.

Imagine a column with 100 rings and 100 trays. Imagine a scenario wherein the 100 trays are going to be replaced by 150 trays. This is sometimes called a "three-for-two revamp" and is shown in Figure 7. One option is to use many of the existing rings and then to weld many new rings to the inside of the column [*4*]. This welding will necessitate heat treating. Another option is to support new rings using old rings and vertical posts. As shown in Figure 8, FRI demonstrated the post-supporting strategy of new rings in one of its test towers. This strategy avoids the necessity for heat treating, because the new rings and posts are not welded directly to the shell of the pressure vessel.

Referring back to Figure 6, most columns contain manholes. These manholes allow humans to enter columns for inspection, maintenance and revamp work. Figure 9 is a photograph (side view) of one of FRI's manholes. The inside dimensions (IDs) of column manholes are of extreme importance when planning a revamp and when designing parts for trays and for packing distributors. The parts must, of course, fit through the manhole, and then the parts must be "rotate-able" into vertical orientations, for transporting downward inside the column. Similarly, large blocks of structured packing must fit inside the open manholes. In some cases, large parts of trays and distributors are broken down, in the planning and drawing stages, into sub-parts. For example, a large central tray downcomer might be designed and fabricated as two pieces. In some cases, column-revamp work is facilitated by cutting the tops off of columns and then reattaching those tops after the revamp work is completed. Very unfortunately, the history of global column-revamp work includes numerous cases where tray and packing parts did not fit into columns after those parts were designed, fabricated and delivered to revamp sites.

Levelness

Some old and new columns are not perfectly straight, or upright. Long column shells are fabricated in pieces. Sometimes when the shell pieces are welded together, the column makes a slight turn. Additionally, long shells can become bowed, especially if left in a horizontal position in the sun for too long. Once a long column is erected, that is, uprighted, it is difficult to see if the column is bowed, especially because of the column's insulation, ladders, platforms and piping hook-ups. On the other hand, if a column is not perfectly upright, that can often be seen from about 100 yards away. A column that is off vertical by only 1 deg can be seen by eye.

Columns that are not perfectly vertical usually have rings that are not perfectly horizontal. When trays or packing distributors are placed upon those rings, those trays and distributors are prone to malperformances — sometimes not meeting process targets. It is also, of course, possible that the rings were installed improperly in the shell fabrication shops. Tray and packing vendors, engineering companies and operating companies all have standards regarding ring levelness tolerances. Usually, larger tolerances are allowed at larger diameters. It is very important that tray and distributor installation companies adhere to these levelness tolerances, regardless of how difficult they sometimes appear to be. In worst-case scenarios, shims can be employed on the rings to install trays and distributors without significant out-of-levelness. Again, very unfortunately, the history of global column-revamp work contains numerous cases where non-horizontal trays and distributors performed poorly.

Before initiating serious column revamp work, a column should be gamma-scanned especially at processing conditions near its flood point.

Gamma scanning

Before initiating serious column revamp work, a column should be gamma-scanned especially at processing conditions near its flood point. Gamma scanning can identify the location of any hydraulic bottlenecks in trayed or packed columns. Once the location has been identified, tray and packing engineers, especially from the vendor companies, can determine the reason that the hydraulic bottleneck is occurring. Column flooding is often defined as "liquid cannot get down the column." When column flooding occurs, there are many possible reasons including excessive vapor traffic and excessive liquid traffic. Column flooding usually initiates at a single vertical point in a column. Because the full liquid stream is not getting past that point, the section of the column beneath the bottleneck is often starved of liquid. At the same time, the section of the column above the bottleneck is accumulating liquid. This starvation and accumulation are observable in thorough top-to-bottom gamma scans. Reboiler and condenser circuitry should also be scanned at those rates where a column appears to be flooded. Also, premature flooding is often attributable to solids and plugging. Sometimes, column internals can simply be cleaned, rather than replaced.

Larger columns

In 1985, the number of global columns over 300 ft in height could have been counted on one hand. Today, several global columns are approaching 350 ft in height.

In 1985, a propylene column 15 ft in diameter was considered to be "huge." Today, pro-

Source: FRI

FIGURE 9. Column manholes allow humans and equipment to enter

pylene columns 30 ft in diameter have been commissioned and are being designed.

A column that is 30 ft in diameter has four times the cross-sectional area of a 15-ft-dia. column. As a result, with large-diameter trayed and packed towers, the possibilities for liquid and vapor maldistributions are greatly increased. With trays, for example, long liquid-flow path lengths help to create hydraulic gradients in the froths. In response to the froth height differences, vapor streams have a natural tendency to flow through the deck areas having the shortest froth heights. As a worst-case scenario, vapor crossflow channeling (VCC) can occur [*5*] and tray efficiencies can suffer.

Again, very large trays have very large deck areas. Such areas are particularly prone to froth stagnations, or even retrograde (backwards) froth flow. If properly designed and specified, push valves on those deck areas can keep the froths moving toward the outlet weirs.

Regarding packing, the difficulties associated with the attainment of perfect liquid distributions at the tops of packed beds are increased appreciably with very large column cross-sectional areas. Some companies, including vendors, insist that all liquid distributors be tested with water at vendor company test stands.

The bottom line is that, with trayed and packed towers, malperformances are more likely to occur before and after column revamp work, when tower diameters are extremely large.

Very large and very tall columns bring up other issues. Revamping such towers during 30-day turnarounds is often excessively challenging. A multiplicity of teams, and work elevations, are required. Each team requires an observer/coordinator at each manhole and also its own on-the-ground assistants to keep parts moving down and up the tower.

Tide marks

As trays and packings are being removed from a tower, those trays, packings and packing distributors need to be examined before they are discarded or reused. Besides plugging, one of the first things to look for is metal thinning due to corrosion. Some services are particularly prone to acid build-ups, and acids eat away at metals, including column shells. Sometimes, so-called “tide marks” can be seen on the inside walls of trayed columns that have been running for many years. Those tide marks show froth heights, and maybe froth height differences. Figure 10 is a photograph from one of FRI’s kettle reboilers [*6*]. Tide marks are clearly evident on the inside walls of that kettle. In this case, those tide marks indicate the average depth of the boiling pool.

Final post-revamp inspections

After any revamp, there comes a point in time when a column is ready for a final inspection, before the manhole covers are closed and the column is buttoned up. This inspection should always involve a member of the company that owns and operates the column.

Trays are usually equipped with manways that allow workers and inspectors to climb vertically through tray stacks. Multi-pass crossflow trays, such as four-pass and six-pass trays, are usually equipped with multiple vertical manway paths. The downcomers and anti-jump baffles of multi-pass trays make it impossible to inspect an entire freshly installed tray from just a single set of vertical manways. Such multi-pass trays require that each set of vertical manways be climbed through so that every area and volume of a freshly installed tray can be inspected before those manways and column manholes are closed.

It is, of course, impossible to climb vertically through a bed of packing, whether random or structured. Nevertheless, it often happens that liquid distributors are positioned at the same elevations as column manholes. It is therefore easily possible to inspect most packing distributors before the manholes are buttoned up.

All findings of column inspections need to be thoroughly documented, including inspector names and dates.

Safety considerations

General. There are many people who perform column revamps very regularly. There are other people who become involved in such work only occasionally, or even just once in a career. These people include engineers, technicians, mechanics, welders, electricians and so on. The next few paragraphs are particularly for the people who are not already experienced, and expert, at column revamp safety aspects.

Ultimately, *you* are responsible for your own safety. At the plant, you are in your shoes. Only you go where you go. You should not expect to have an experienced safety expert at your side everywhere that you go at a plant during a column revamp.

The first people to talk to regarding column revamp safety are the safety professionals and the experienced veterans (engineers and technicians) where you work. Your company has, or should have, procedures and policies regarding plant visits. Training classes will possibly be needed. Paperwork will need to be attended to, earnestly. A second group of people to talk to are the safety professionals and experienced veterans at the plant. This includes engineers, outside technicians and board operators. On an increasing basis, plants are providing safety training to first-time entrants to those plants. This training can last an hour or as long as three days. Sometimes, tests will need to be taken at the plant, to assure everybody that you are fully ready to encounter all of the known and potential hazards that are present near and in columns during revamps.

OSHA standards. There are several applicable federal Occupational Safety and Health Administration (OSHA; Washington, D.C.) standards. Certain states and localities have their own standards and these must, and should, be adhered to also. Among the applicable federal standards are the following:

OSHA Standard 29 CFR 1910.146 applies to entry into confined spaces. During a column revamp, some people need to enter the column. Those people need to know what was processed in the column. Were there any poisons or dangerous hydrocarbons or chemicals? Is the column properly blanked off so that those chemicals have no possibility to reenter the column? Has the appropriate equipment been locked out and tagged out (LOTO)? It should be assumed, by the way, that all valves leak. Has the column been steamed out? Aired out? Is the oxygen content going to be monitored continuously while the vessel is occupied?

Regarding entry to any confined space, a key player is the attendant, sometimes called the "hole watch," who is usually positioned outside of any manhole where people enter the column. Nobody enters the column without the attendant's full permission and knowledge. The attendant logs all entries and exits. The attendant has many other responsibilities, including the blowing of an air horn if any dangers arise outside of the column, and, the contacting of rescue personnel if anything goes wrong inside the column. The attendant must sometimes prevent the rescue team from rushing into the column if the rescue team might possibly suffer the same calamity as what brought the team to the column in the first place. Very unfortunately, a large fraction of confined-space rescues involve fatalities and the rescue teams end up focusing on body retrievals. Most importantly, the attendant should be regarded as "the boss."

Falls are the most common cause of work-related injuries and deaths.

OSHA Standard 29 CFR 1910 also includes chapters regarding falls and fall-protection equipment. Falls are the most common cause of work-related injuries and deaths. Climbing up and down the outside of a column involves the risk of falling. Climbing up and down inside the column involves the same risk. As was stated previously, some of these newer columns are 350 ft tall. Body belts, harnesses, deceleration devices, lanyards, lifelines, arrest systems, ropes, ladders and scaffolds are all available to make the climbs safe.

OSHA Standard 1910.132 applies to personal protective equipment (PPE). For column revamp work, the following are regarded as possibly applicable PPE: hard hats, gloves, safety glasses with side shields, steel-toed boots, shields, barriers, respiratory devices and flame-resistant clothing. Anybody who wears PPE needs to be trained regarding the PPE. PPE should always be regarded as the last line of defense against injury. To avoid PPE coming into play at a revamp site, engineering controls, policies and procedures need to be in place, and adhered to. For example, platforms on the sides of columns have guardrails and toe guards. These are intended to prevent people or untethered

Source: FRI

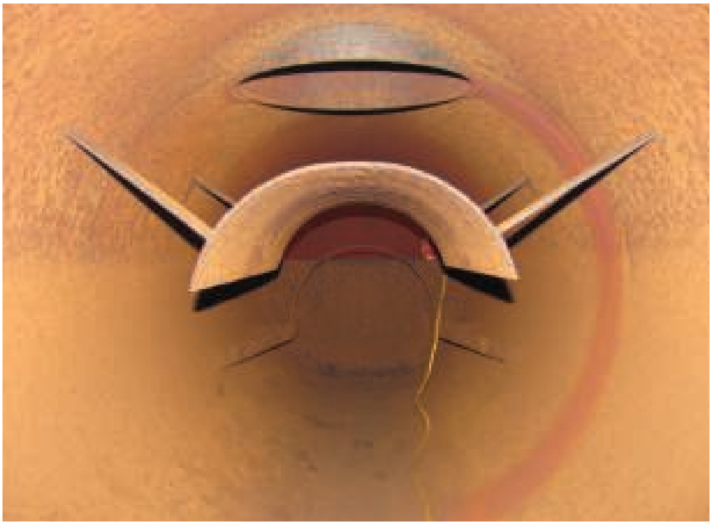


FIGURE 10. Tide marks can sometimes be found inside of old vessels, in this case, a kettle reboiler

equipment from falling off of the platforms. If equipment does somehow fall from a platform, hard hats being worn by the people underneath will lessen the injuries, if any, sustained by those people. The first line of defense is the guard rails and the toe guards.

Safety – policies and procedures. LOTO stands for "lock out tag out" and is a policy and a procedure whereby energy sources (potential, kinetic, pneumatic, radiation, electric and so on) are totally isolated from humans. Dangerous machines are shut off and cannot be restarted easily. Hazardous power sources are isolated and made inoperative often via padlocks. Associated with those padlocks are cardboard or plastic tags that are easily seen and that identify the source of the padlocks. When two or more companies or subcontractors need to be protected from an accidental restart of those machines, it is sometimes required that those two or more companies each add their own padlocks to the machine.

JHA stands for "job hazard analysis" and can be applied to any small or large project. Via a JHA, the hazards associated with a project are identified. The means by which the dangers are eliminated, or reduced to reasonable levels, are listed and enacted. Most JHAs ultimately involve at least one multi-person meeting where the listed hazards and actions are discussed openly.

MOC stands for "memorandum of change." Any change to a unit's piping and instrumentation diagram (P&ID) or procedures needs to be preceded by an MOC, which should also involve an open discussion between many parties. MOCs, including the appropriate costs, ultimately need to be approved by management before a change or changes are actually effected. Once such changes have occurred, the unit's process flow diagrams (PFDs) and P&IDs need to be updated to include the changes. All operating units must have up-to-date PFDs and P&IDs while those units are operating.

PSSR stand for "pre-startup safety review." After a unit has been modified, and before the blinds are removed, the valves are opened and the pumps are restarted, a meeting should be held to determine whether the unit is truly totally ready to be placed back into service. Among the questions that need to be answered is this one: Have all affected employees been apprised or trained regarding the changes that have been made to the unit?

Miscellaneous. Column owners or operators rarely do their own revamp work. Some vendors of tower internals have field service organizations at their disposal. Otherwise, there are very experienced companies that perform such work.

Before any column can be entered by humans it must be steamed-out, especially to remove chemicals that adhere to the column wall and the column internals — subsequent to the pump-out. Every column and service is different. Some columns are steamed for 24 h, whereas others require three full days. Thereafter, air movers are required to establish breathable atmospheres and to prevent air from becoming dangerously stale. Thereafter, the column is still not ready for full entry. Workers with respiratory equipment should peer carefully into the column and take samples of any liquids or solids (for instance, deposits) that are found. Those samples require analyses. For example, pyrophoric compounds, such as polybutadiene, are sometimes in evidence in olefin plant depropanizers, even after long steam-outs. It sometimes takes a full week, after shutdown, for some columns to be totally ready for human entries.

Column revamp work, especially for those people who do not get involved in it routinely, is very tiring. For example, when climbing up the outside of a column, a worker's arms become fatigued before the legs. On very rare occasions, with very tall columns, special scaffolds are erected and those special scaffolds are equipped with elevators. Revamp workers who have such elevators at their disposal should consider themselves lucky.

When first entering any plant, notice should be taken of the wind sock and the wind direction. If anything goes wrong in a nearby operating unit, know where to go to avoid any harmful vapors, including smoke.

Column revamps can occur anywhere and

during any season. Dress appropriately. Wind speeds near the tops of columns can easily be twice those at ground level. Wherever you go when you are at a plant, somebody must know where you are.

Whenever two or more teams are working at different elevations inside a column, means must be in place to assure that workers lower in the column are not struck by objects that fall by accident from above. The fact that all of the people inside the column are wearing hard hats is insufficient. Nets can be strung horizontally inside columns to protect the lower workers.

New plants have manholes that are nominally 24 in. in diameter. Older plants often have manholes that have IDs of only 17 in. Some humans simply will not fit through such small manholes, especially when they are wearing full safety gear. Then, even if those people fit through those manholes, rescue difficulties are exacerbated.

People who enter columns, and especially tall columns, for the first time sometimes become petrified — and even dysfunctional. Attendants must keep an eye out for such people and such people should not be allowed to enter columns until they can demonstrate that their fears are under control.

Structured packing has been known to catch fire in dozens of columns [*7*]. Usually, sparks and slag from welding ignite deposits on the structured packing sheets and then the burning spreads to the sheet metal. Once these fires begin, they are very difficult to stop. Some revamp companies and some operating companies insist that structured packings be removed from a column before any welding work is performed above those packings.

General. Very unfortunately, some injuries are permanent. Like they said at IBM — think! Like one of DuPont's safety programs — Take Two minutes before embarking on even the smallest of new adventures. Seek the advice and assistance of safety professionals and experienced veterans, including board operators and outside technicians. Some of those people have made safety-related mistakes and are eager to tell you about those mistakes. While at the plant, never be lulled into a false sense of security. Expect the unexpected — and "unexpect" the expected.

It has been estimated that there are 100,000 columns in the world. Many of them have already been revamped. All of them are targets for potential future revamps. If you become involved in one, or some, of those revamps, be totally prepared. Be safe. ■

Edited by Gerald Ondrey

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Part 1

Vacuum Drying: Basics and Application

Vacuum drying can be a useful tool for solid products that are heat-sensitive. Here are some guidelines for the selection and use of various types of vacuum dryers

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DPharma Group

IN BRIEF

Drying is an essential unit operation in a variety of chemical process industries (CPI) sectors. Food, pharmaceutical, chemical, plastic, timber, paper and other industries use drying equipment to eliminate moisture during product processing. Most dryers are classified as direct dryers, where hot air (at near atmospheric pressure) is used to supply the heat to evaporate water or other solvents from the product. Another important dryer category, vacuum dryers, involves the use of a reduced-pressure atmosphere to surround the product.

Drying is among the most energy-intensive unit operations, due to the high latent heat of vaporization of water and the inherent inefficiency of using hot air as the (most common) drying medium. Depending on the specific product attributes required, different industry sectors require different types of drying technology. Drying high-value products that are likely to be heat-sensitive, such as food, pharmaceuticals and biological products, demands special attention. When dried by convection at higher temperatures, these heat-sensitive products degrade, change color and appearance and have lower vitamin or nutrient content. Vacuum dryers offer an alternate path. This article discusses the operation and selection of vacuum dryers, and provides examples of applications in which vacuum drying is used.

Drying principles

Drying involves two distinct drying periods, known as the constant drying period and the falling drying period (Figure 1). Drying occurs when liquid is vaporized by supplying heat to the wet feedstock. The liquid removed by the drying process could be either free moisture (unbound) or bound within the structure of the solid. The unbound moisture, normally present as a liquid film on the surface of a solid particle, is easily evaporated, while the bound moisture could be found within the solid material, trapped in the microstructure of the solid. In this case, the moisture must travel to the surface to be evaporated. When a solid product is subjected to drying, removal of unbound and bound moisture depends on the rates at which these two processes proceed. Removal of unbound moisture depends on external conditions of air or gas temperature, flow, humidity, area of exposed surface and pressure. The movement of bound moisture depends on the nature of the product being dried and the extent of moisture within the product.

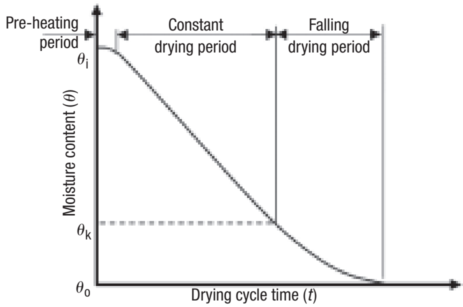

FIGURE 1: Drying processes involve two distinct phases

Unbound moisture normally is removed by evaporation and vaporization. Raising the temperature facilitates the evaporation and air draws the moisture away. If the product being dried is heat-sensitive, then the temperature at which evaporation occurs (at the boiling point of water or other solvent) can be reduced by lowering the pressure with a vacuum.

Vacuum-drying advantages

Vacuum drying is a viable technology that has been used successfully for many years in the pharmaceutical, food, plastic and textile industries, among others in the CPI.

TABLE 1. SELECTION CRITERIA FOR THE INDIRECT DRYERS [1]								
Dryer type	**Plate**	**Drum**	**Tumbling**	**Vibrating**	**Conical**	**Thin film**	**Paddle**	**Mixer-kneader**
Requirements								
Continuous	1	1		1	4	1	1	1
Discontinuous	4	4	1	4	1		1	1
Vacuum	1		2		1	1	1	1
Large surface area and volume	2	3	3	3	2	1	2	1
High specific capacity	2	1	3	1	2	1	2	1
Materials								
Friable	1	4	1	1	1	2	1	1
Fluid	4	1	4	4	4	1	3	1
Viscous/pasty	4	1	4	4	4	4	3	1
Crusty	4	4	4	4	4	3	2	1
Processing								
Mechanical	1	1	1	3	1	2	2	2
Thermal	2	3	3	2	2	3	3	3
1 = ideal, 2 = good, 3 = sometimes suitable, 4 = not suitable								

A major advantage to vacuum drying is its energy conservation — less energy is needed for drying, cutting down on the economic and environmental costs associated with drying a product for storage, sale or other purposes. Vacuum-drying processes also tend to work faster than other drying methods, cutting down on processing times, which can be important in some facilities where products are being moved through quickly. Another advantage of drying materials in this way is a less damaging drying process. Some materials can experience problems at high temperatures, such as developing hard, leathery crusts from heat exposure during the drying process. Vacuum drying tends to retain the integrity of the original item without damaging it with heat. For foods and pharmaceuticals, this can be valuable, as other drying processes can degrade quality and make the food less appealing or affect potency of heat-sensitive drug products.

Using vacuum-drying equipment also reduces risks to workers. With other types of drying equipment, there are vented fumes and particles that can potentially be hazardous and require operators to wear personal protective equipment. With a vacuum dryer, ventilation does not occur, and personnel working near the dryer are safer. It is also possible to recover the precipitated moisture collected during the drying for further use.

Vacuum-drying operation

The majority of dryers are of the direct (or convective) type, where hot air is used both to supply the heat for evaporation and to carry away the evaporated moisture from the product. Notable exceptions are freeze and vacuum dryers, which are used almost exclusively for drying heat-sensitive products. Vacuum dryers tend to be significantly more expensive than dryers that operate near atmospheric pressure.

Vacuum drying is a process in which materials are dried in a reduced pressure environment, which lowers the heat needed for rapid drying. Vacuum dryers offer low-temperature drying of thermolabile materials and are suitable for solvent recovery from solid products containing solvents. Heat is usually supplied by passing steam or hot water through hollow shelves (Figure 2). Drying temperatures can be carefully controlled and, for the major part of the drying cycle, the material remains at the boiling point of the wetting agent. Drying times are long, usually about 12 to 48 h. Unlike a direct-heat dryer — in which the material is immersed directly into the heating media (usually a hot gas

FIGURE 2: Vacuum tray dryers are common for laboratory and pilot-scale work

FIGURE 3: Traditional blenders can be modified to be used as vacuum dryers (Source Patterson Kelley)

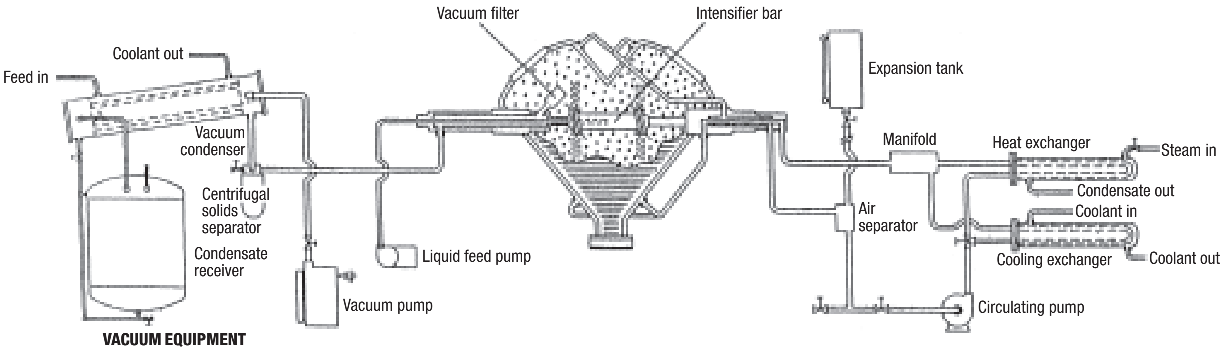

FIGURE 4: Vacuum-drying systems for solvent-based products employ condensers to collect solvents (Source: Patterson Kelley)

stream) and is dried by convection — a vacuum dryer is an indirect-heat dryer (Table 1). That is, the heat is transferred to the material as it contacts the dryer's heated surface, drying the material by conduction.

Understanding this distinction is essential for grasping the advantages and limitations of vacuum drying, as well as for selecting a vacuum dryer that efficiently and economically achieves process goals.

To understand how vacuum operation can aid drying, consider the following equation, which represents a simplified drying theory:

$$Q = U\,A\,\Delta T \quad (1)$$

Where Q is the total heat (in British thermal units, Btu), U is the overall heat-transfer coefficient (in Btu/ft^2/°F), A is the effective heat transfer surface area (in ft^2), and ΔT is the temperature difference between the liquid's boiling point (that is, its vaporization temperature) and the heating media's temperature (in °F). The process goal is to achieve an effective heat transfer (Q) to the material so that its liquid content is vaporized.

Most often, the material's properties and the dryer type effectively establish the U and A values for the process. So the process-efficiency objective should be to maximize the ΔT value, in order to increase the Q value. By controlling atmospheric pressure, the vacuum dryer increases the effective ΔT for a given process. It reduces the boiling point (vaporization temperature) required for removing the liquid.

Effective ΔT can be significantly increased by controlling pressure and heat to the dryer, facilitating faster drying than at normal atmospheric pressure. Hence, heat-sensitive materials such as foods, pharmaceuticals and antibiotics can be dried with vacuum drying with shorter drying times and at lower temperatures. The closed system also offers the advantage of handling reactive compounds or hazardous solvents in the product being dried. The vacuum dryer safely contains and condenses the hazardous vapors from such substances without any threat to the workplace environment or to the outside atmosphere.

Vacuum drying is predominantly operated as a batch unit operation. However, a vacuum-drying unit can also be integrated as part of a continuous process. In those cases, proper control of the feed and discharge materials is critical, along with proper process-control parameters.

Limitations of vacuum dryers are generally related to the heat-transfer mode of the equipment. A vacuum dryer's upper temperature limit (typically about 600°F) is lower than that of a direct-heat dryer. The rate at which material temperature can be raised in a vacuum dryer is also limited. This is because the indirect-heat dryer is limited by the surface area available for heat transfer, unlike a direct-heat dryer, which is limited only by the hot-gas volume in the drying chamber. The vacuum pump is primarily responsible for the vacuum level inside the dryer.

Vacuum drying options

Most vacuum dryers are adapted from solids blenders. The two principal types of vacuum dryers are tumble and agitated. A number of traditional blenders can be modified for use as vacuum processors (Figures 3, 4 and 5). Guidelines for selecting general and vacuum dryers are given in Tables 1–3.

Tray dryers. The most common dryer for laboratory and small-scale

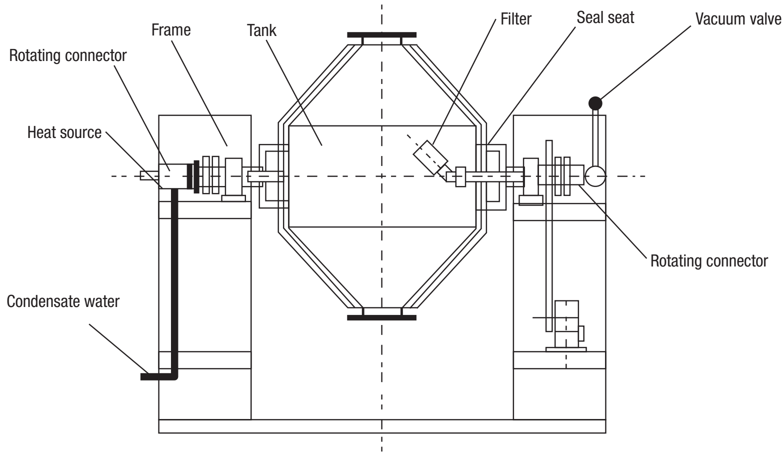

FIGURE 5: This double-cone blender is another example of modified vacuum dryer (Source: Paul O. Abbé)

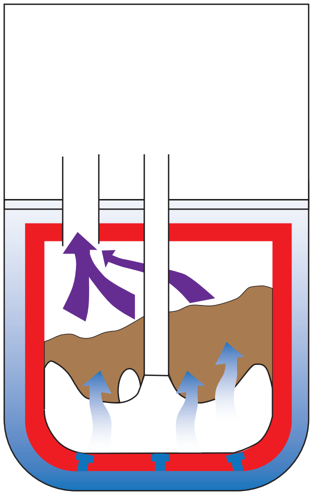

FIGURE 6: Inert gas can be passed through products to assist solvent removal in vacuum dryers (Source: GEA Pharma systems)

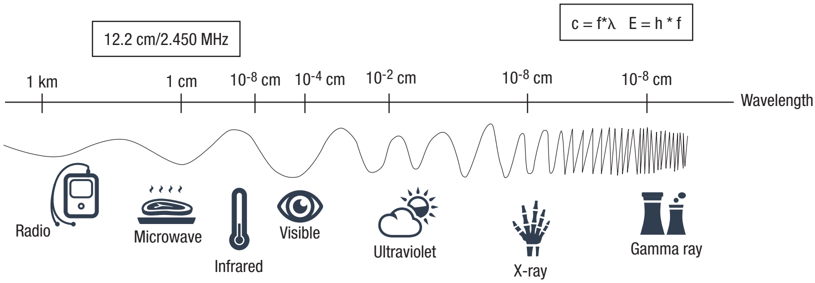

FIGURE 7: Microwave radiation frequencies range from 300 GHz to 300 MHz

pilot work is the vacuum tray dryer (Figure 2). Heat transfer in this type is largely by conduction or by radiation. The trays are enclosed in a large cabinet, which is evacuated. The water vapor produced is generally condensed, so that the vacuum pumps have only to deal with non-condensable gases. Tray dryers are reliable and have no moving parts, but operation and cleaning are labor-intensive. Also, because solvent wicking can cause a crust to form on the cake, the product often requires milling, screening, blending or other post-drying treatment to ensure homogeneity. These problems can be avoided by keeping the cake moving during drying, which is the major advantage of cone, paddle and tumble dryers. Vacuum tray dryers consist of a main body completely heated by a liquid circulation circuit, so as to avoid any condensation phenomena, and by a series of shelves heated by means of a fluid distribution collector, which guarantees heat homogeneity on all radiating plates. The inner walls and the shelves are perfectly polished.

The shelves are heated inside the vacuum chamber. This technique can apply heat indirectly to the product by forcing physical contact with the shelf. A hot medium flows through the shelves, thus enabling it to conduct heat to the tray, which is positioned on the shelves.

Granulators. Traditional high-shear granulators in the pharmaceutical industry are modified to be vacuum processors by using vacuum to pass heated air or inert gas through the product and to transport solvent vapors out of the processor. In flow-through processors (Figure 6), drying is accomplished by passing the heated gas through the product to remove solvents using vacuum. By incorporating condenser systems and selecting the right pumps, excellent solvent recovery and a competitive drying rate can be achieved. The bowl can be swung to provide gentle turning of product while undergoing drying under vacuum.

Microwave vacuum drying. Microwaves are a form of electromagnetic radiation with frequencies ranging from 300 GHz to 300 MHz (Figure 7). Microwave drying refers to the use of the dielectric heating principle, which relies on high-frequency electromagnetic oscillations caused by molecular motion. The mechanism for energy transfer during microwave heating is delivered directly to materials by molecular interactions with an electromagnetic field and the conversion of electrical field energy into thermal energy.

Microwave drying (Figures 9 and 10) has significant advantages compared with conventional drying, because in microwave drying, heat is generated by directly transforming the electromagnetic energy into kinetic molecular energy. Thus, the heat is generated deep within the material to be dried. Especially in microwave vacuum drying, this approach has significant advantages for bulk products with poor thermal conductivity. Microwave drying is comparatively the fastest drying method available in single-pot systems (Figure 8).

Because microwave heating is a form of dielectric heating, the materials' dielectric properties are thus the most important factors. The molecules of such substances form electric dipoles which, when exposed to an electric field, assume an orientation relative to the direction of the field. It is this orientation polarization that is responsible for generating energy.

In the rapidly alternating electric field generated by microwaves, polar materials orient and reorient themselves according to the direction of the field. With the rapid change in the field at 2,450 MHz, the orientation of the field changes 2,450 million times per second and causes rapid re-orientation of the molecules, resulting in increased kinetic energy, friction and heat creation.

Freeze drying. Freeze-drying, also known as lyophilization, is a dehydration process typically used to preserve a perishable material or make the material more convenient for transport. Freeze-drying works by freezing the material and then reducing the surrounding pressure to allow the frozen water in the material to sublimate directly from the solid phase to the gas phase.

Freeze-drying is divided into three phases:

1. An initial freezing process, carried out such that the product exhibits the desired crystalline structure. The product is frozen below its eutectic temperature (the highest allowable product temperature during the conditions of sublimation)
2. A primary drying (sublimation) phase during which the partial pressure of the vapor surrounding the product must be lower than

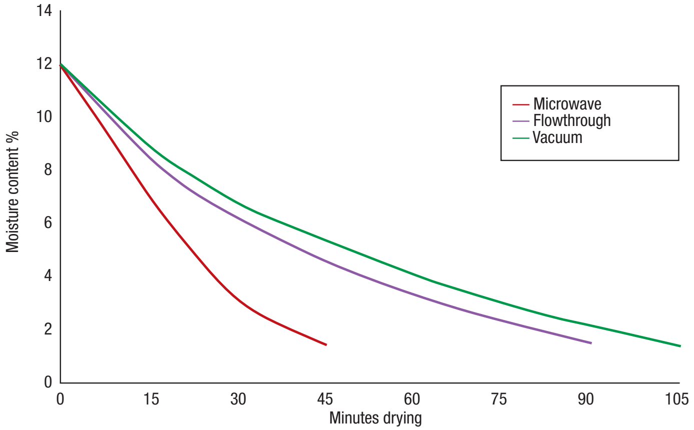

FIGURE 8: Microwave-assisted vacuum dryers can achieve faster drying times compared to vacuum alone and inert gas with vacuum (Source: GEA Pharma Systems)

the pressure of the vapor from the ice, at the same temperature. The energy supplied in the form of heat must remain lower than the product's eutectic temperature

3. A secondary drying phase aimed at eliminating the final traces of water, where the partial pressure of the vapor rising from the product will be at its lowest levels

Hybrid technologies

Hybrid, or combined drying technologies involve implementation of different modes of heat transfer and two or more stages of the same or different type of dryer [*2*]. The efficiency of drying, in terms of both energy and process duration, is an area of intense research, and some of the most promising drying methods include the use of electromagnetic waves and sonic-wave-assisted drying.

Applications

The following describes examples of areas where vacuum-drying technologies are employed and are being explored further.

Vacuum drying pharmaceuticals. The preparation of mouth-dissolving tablets provides an example of the usefulness of vacuum-drying techniques. In this case, mouth-dissolving tablets were formulated from granules containing Nimesulide, camphor, crospovidone and lactose using a wet-granulation technique. The granulation step was followed by a tablet-compression step. When vacuum drying was applied during the granulation stage, the subsequently formed tablets were of higher quality than those where the vacuum was applied to the already compressed tablets. The scientists in this case concluded that the vacuum-drying technique would be an effective alternative approach compared with the use of more expensive adjuvants in the formulation of mouth-dissolving tablets [*3*].

Drying bacteria. Normally, probiotic bacterial strains and starter cultures are dry-frozen to preserve them until use. That means, they are first deep-frozen and afterward, dehydrated in a vacuum. This procedure has two major disadvantages in practice: First, it consumes a very high amount of energy; and second, some bacterial strains do not survive temperatures below 0°C. The Technical University of Munich (www.tum.de) has developed low-temperature vacuum drying (LTVD) for industrial processes. LTVD can dry unstable substances at moderate temperatures above zero without causing too much damage to the cell structure.

Vaccines and other injectables. Freeze-drying is routinely employed to produce pharmaceutical products [*5*]. Pharmaceutical companies often use freeze-drying to increase the shelf life of products, such as vaccines and other injectables. By removing water from the material and sealing the material in a vial, the material can be easily stored, shipped, and later reconstituted to its original form for injection. Another example from the pharmaceutical industry is the use of freeze-drying to produce tablets or wafers, the advantage of which is that less excipient material is required. In addition, freeze-drying allows a rapidly absorbed and easily administered dosage form.

Food industry. As mentioned above, reasons for the growing interest in microwave heating can be found in its peculiar mechanism for energy transfer: during microwave heating, energy is delivered directly to materials through molecular interactions with an electromagnetic field via conversion of electrical field energy into thermal energy [*6, 7, 8*]. This can allow unique benefits, such as high efficiency of energy conversion and shorter processing times,

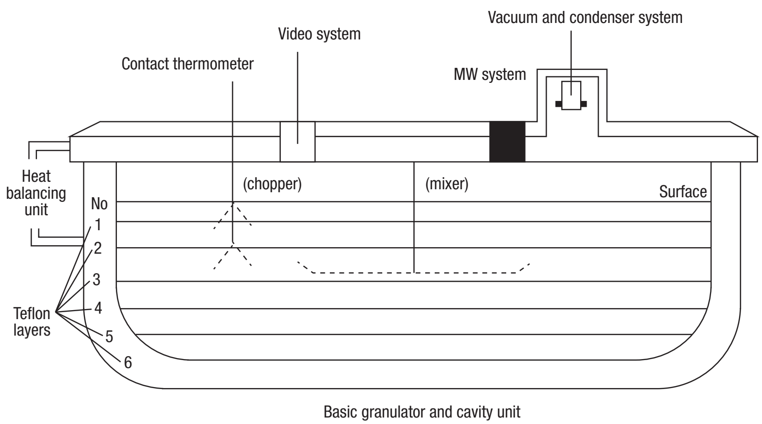

FIGURE 9: Microwave vacuum drying at production scale, as shown in this schematic diagram of a microwave vacuum dryer, depends a great deal on the materials' dielectric properties

FIGURE 10: Microwave vacuum dryers can help avoid damaging food products (Source: Bohle)

TABLE 2. QUANTITATIVE INFORMATION NEEDED TO ARRIVE AT A SUITABLE DRYER [*20*]
• Dryer throughput; mode of feedstock production (batch versus continuous)
• Physical, chemical and biochemical properties of the wet feed, as well as desired product specifications; expected variability in feed characteristics
• Upstream and downstream processing operations
• Moisture content of the feed and product
• Drying kinetics; moist solid sorption isotherms
• Quality parameters (physical, chemical, biochemical)
• Safety aspects (fire hazard and explosion hazards, toxicity)
• Value of the product
• Need for automatic control
• Toxicological properties of the product
• Turndown ratio, flexibility in capacity requirements
• Type and cost of fuel, cost of electricity
• Environmental regulations
• Space in plant

TABLE 3. VACUUM DRYING APPLICATION IN FOOD INDUSTRY

Products	Dryer type	Comment	Reference
Strawberries and carrots	Microwave	Preliminary drying studies	11
Pistachios	Vacuum-infrared	modeling	12
Eggplants	Vacuum	Drying characteristics studies	13
Probiotics	Ultrasonic vacuum spray dryer	Making highly viable probiotics	14
Chilis	Combined microwave-vacuum rotary drum dryer	Experimental studies	15
Cranberries	Microwave-vacuum dryer	Energy use and efficiency studies	9
Cranberries	Microwave-vacuum dryer	Evaluation of quality	9A
Fruits and vegetables	Microwave vacuum dryer	Drying systems in agricultural production	16
Food (general)	Microwave	Microwave applications in thermal food processing	17

thus leading to reductions in manufacturing costs due to energy saving (microwave heating can be a tool of process intensification). By combining vacuum with microwave as the source of thermal energy, a product is dried faster and at a lower temperature, thus avoiding damage to the product.

By using pulsed-microwave vacuum drying, researchers have been able to dry cranberries [*9*]. Researchers have microwave-vacuum-dried mint leaves and compared the process with air drying [*10*]. The effective moisture diffusivity was significantly increased when microwave drying was applied under vacuum conditions, compared with hot-air drying. For color, the microwave-vacuum-dried mint leaves were light green/ yellow, whereas the hot-air-dried mint leaves were dark brown.

Plastics production. The plastics industry uses vacuum to remove moisture from engineered plastics. If the moisture is not extracted from pellets before melt processing, streaks, bubbles, burning, brittleness and other critical defects in the molded or extruded parts can occur.

Emerging areas. Recently, there has been an increased level of interest in developing drying technologies as alternatives to lyophilization in the formulation of proteins as dry powders. Mannitol is often added to dried protein formulations as the bulking agent because it has the tendency to crystallize rapidly from aqueous solutions.

Vacuum-drying hybrid technologies are being investigated to overcome some of the issues associated with the process of lyophilization. These limitations include long processing times (typically 3–5 d), expensive setup and maintenance of the lyophilization plants, and, most of all, the steps inherent in freeze-drying can lead to instabilities in the protein structure. Due to the complex structural properties, proteins have a tendency to denature and undergo irreversible aggregation during various processing steps of drying [*4*].

Optimizing drying processes

To optimize vacuum-dryer performance, it is important to adjust the peripheral equipment to match the specific needs of the drying operation. This equipment includes the dryers' heating and cooling system, dust filters, condensers and vacuum pumps. Condensers are used mostly to recover process solvents, which are evaporated during drying. They are typically shell-and-tube type surface condensers, arranged either vertically or horizontally. Condensate collection can be measured and combined with a mass balance to allow indirect, realtime monitoring of product moisture during the drying process.

Drying efficiency. To increase the efficiency of drying processes, emphasis has recently been placed on the development of new technologies that use alternative sources of energy to enhance the heat transfer between the product and heat source (for example, microwave, radiofrequency and infrared radiation). New technologies can also intensify

TABLE 4. SUGGESTED VACUUM DRYER SELECTION APPROACH [*21*]	
1	What is the batch size you want to process?
2	Consider which dryer type will drive up the temperature difference between your material and the heating media (*ΔT*)
3	Determine the moisture (or liquid) content of your product to be dried, as well as the nature of that liquid (water or solvents)
4	Determine bound liquid and un-bound liquid is in the product
5	Based on the liquid, determine the vapor pressure profile with the temperature to be used
6	What is final product specification regarding percentage of liquid remaining in the product?
7	What utilities are available in the plant?
8	Consult a reputable dryer manufacture, discuss the product and determine the budget
9	Perform tests on your product in the dryer manufacturer's facility

the dehydration rate without increasing the amount of heat supplied to the product (for example, organic solvents, ultrasonic waves). Some of these technologies have already been developed and tested for use in freeze-drying foods [*18*].

Selection of dryers. Determining which vacuum dryer is best for a particular application (Table 4) depends in part on knowing a solid material's moisture content. In addition, it is important to understand the material's particle characteristics, because these characteristics can vary in unexpected ways at different moisture levels. For instance, a filter cake containing 40% moisture can flow better than one with 15% moisture. For this reason, end-users of dryer technology should expect dryer manufacturers to test solid materials before determining which dryer type is best capable of handling it.

Several dryer types (or drying systems) may be equally suited (technically and economically) for a given application. A careful evaluation of as many of the possible factors affecting the selection will help reduce the number of options. For a new application (a new product or new process), it is important to follow a careful procedure leading to the choice of dryers.

Vacuum operation also eases the recovery of solvents by direct condensation, thus alleviating possible serious environmental problems. Dust recovery is simpler, so that vacuum dryers are especially suited for drying toxic, dusty products, which must not be entrained in gases. Furthermore, vacuum operation lowers the boiling point of the liquid being removed, thus allowing the drying of heat-sensitive solids at relatively fast rates. Dryer selection can have a major impact on product quality, particularly in the case of thermally sensitive materials, such as foodstuffs.

Fuzzy logic. The use of a fuzzy expert system as an aid in the preliminary selection of a batch dryer can be a helpful tool. This incorporates a novel "multiple goal" approach in which different facets, such as dryer type, single versus multiple dryers, and atmospheric versus vacuum operation, are determined independently [*19*]. ■

Edited by Scott Jenkins

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Part 2

Wastewater Sludge Centrifugation Before Drying

The decanter centrifuge is an important piece of equipment for sludge volume reduction prior to thermal drying. Understanding centrifuge operation helps manage drying energy costs

Bart Peeters
Monsanto Europe

IN BRIEF

DECANTER CENTRIFUGE

LAB SIMULATION OF COMPACTION

GOVERNING PARAMETERS

COMBINED SYSTEM

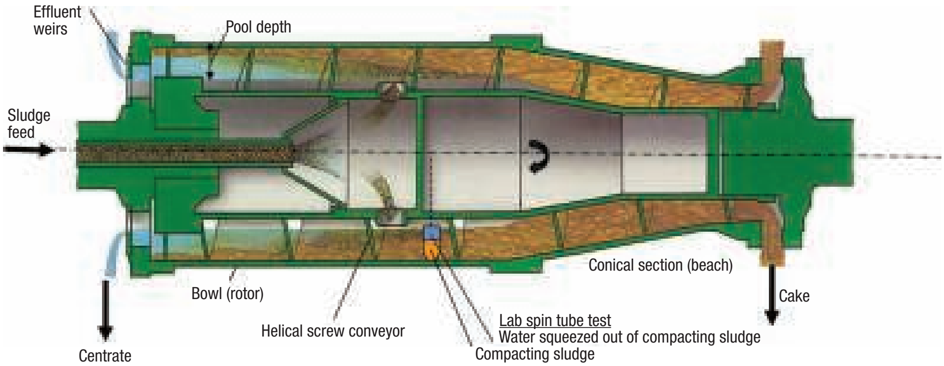

FIGURE 1. This schematic diagram shows a sludge decanter centrifuge (adapted from Andritz), with a spin tube — used in the laboratory to simulate cake compaction — superimposed

Consecutive mechanical dewatering and thermal drying are integral parts of sludge management, lowering the sludge volume that needs to be disposed of or further treated. Because drying is an energy-intensive unit operation, the mechanical pre-concentration of the solids is a prerequisite.

The economic necessity for sludge-volume reduction in the chemical process industries (CPI) as a way to manage downstream sludge-handling costs was discussed previously (*Chem. Eng.*, Sept. 2014, pp. 51–54 [*1*]). Because dewatering prior to drying is such an important stage in the sludge-volume-reduction process — the drier the sludge solids, the less costly the next drying stage — this article discusses sludge dewatering with a solid-bowl decanter centrifuge, one of the most frequently used types of equipment for mechanical dewatering of sludge.

The high energy consumption of sludge dryers is primarily due to the latent heat of evaporation of water, which is equal to 2,260 kJ/kg at 100 °C. To first raise the water temperature from, for instance, 25°C to 100°C, an additional 314 kJ/kg is required. This means that at 100% efficiency, the minimum heat requirement to evaporate the sludge's water is 2,574 kJ/kg or 715 kWh/m^3 of evaporated water. In industrial dryers, the specific energy consumption varies between the latter and 1,400 kWh/m^3 water evaporated [*2*]. The specific power consumption, however, for conventional thickening technology is in the order of magnitude of only 1 kWh/m^3 of wastewater slurry treated [*3*], which makes mechanical solid/liquid separation about 1,000 times less energy-intensive than thermal drying. Within the global decanter-centrifuge application market in terms of machines sold, the market share for sludge dewatering is one of the largest with a 35% share in 2000 [*4*].

Decanter centrifuge

The basic idea of centrifugal solid-liquid separation is simple — apply centrifugal force to a mixture of materials with different densities, and the heavier substance (solid, or a denser liquid) will be thrown further outward, resulting in separation [*5*].

The solid-bowl decanter centrifuge used to dewater waste sludge is constructed of an external solid bowl and an internal Archimedean screw conveyor mounted horizontally (Figure 1). Both rotate in the same direction, with the helical screw rotating at a slightly different speed to the bowl (ranging from 1 to 5 rpm), which allows the solids to be transported. The conditioned sludge is pumped via a stationary, central feed pipe into the hollow shaft of the rotating screw conveyor. The sludge moves through ports in the cylindrical (clarification) part of the bowl. Because of the centrifugal field (up to 3,500 times earth's acceleration due to gravity) as result of the spinning bowl (typically in the range from 2,500 to 3,500 rpm) the heavier sludge flocs move radially outward to the inner bowl wall, while the lighter water remains pooled on top of the sedimented solids (Figure 1). The clarified water flows spirally along the helix of the screw conveyor in the direction of the liquid discharge ports. This centrate then overflows the effluent weirs at the cylindrical end of the centrifuge, similar to river water flowing over a dam.

In the meantime, the sedimented sludge solids are continuously scraped by the screw conveyor toward the solids discharge ports at the opposite, conical end of the bowl. The sludge follows a complicated cork-screw motion as it is pushed along the helical channel formed by adjacent conveyor blades [*6*]. During its 10- to 15-minute transport in the machine, the sludge is dewatered in two steps, filtration and expression [*7*]. During the first filtration stage, water is withdrawn from the sludge to the point where the sludge solids start to make contact with one another, resulting in an initial solid pressure and the expression phase starts [*7*]. Throughout the second and more important expression stage, the original loosely packed solid structure is compressed to yield a much denser state as a result of the centrifugal compaction stress [*3*].

The solids in the underlying layers (solids closest to the inner bowl wall) of the cake under formation experience compressive forces due to the pressure from the weight of the overlying solids (the solids closest to the axis of the centrifuge). The compressive stress is transferred downward to the cake layers more adjacent to the inner bowl wall throughout the continuous linked network of solids. In this way, the sludge cake is further consolidated.

When sludge solids closer to the inner bowl wall get more compressed over time, simultaneously, these solids end up at a larger distance from the center of rotation. As a consequence, the rotational radius for the overlying sludge particles grows as well, and, hence, the force they exert on the lower (underlying) solids increases accordingly. This in turn compresses the lower sludge layer again, and so on [*8*]. This dynamic process continues until equilibrium is reached for any height in the compacted sludge cake.

As a result of the cumulative solids stress exerted on the cake layer closest to the inner bowl wall, the percent dry substance (%DS) is highest in this underlying layer, whereas the cake segment at the surface (closest to the axis of the centrifuge) remains less packed and has a significantly (~10%) lower DS [*9*]. On average, the cake discharged typically has a dryness ranging from 20 to 30% DS, depending on the sludge composition.

Concomitantly with the cake compaction, the interstitial water trapped inside the sludge cake is expelled from the dense structure [*7*]. The water in the sludge matrix flows slowly upward through narrow channels, away from the inner bowl wall (that is, in the opposite direction to the movement of the consolidating solids), toward the upper cake layer, which is less compact and consequently more permeable [*8*]. Water removal from the sludge, by increasing the compressive stress on the flocs, resembles squeezing water from a sponge [*7*]. The compaction of the sludge and the resulting expressed water atop the consolidated sludge is illustrated in Figure 2.

The described dewatering mechanism via compaction (for sludge and other compressible biotic materials) is totally different than the deliquoring mechanism via drainage of drainable solids (such as PVC, PVAC or PVOH polymer grains). In these applications, the pool of liquid lies significantly below the solids-spillover point in contrast to the case of sludge dewatering where a large pool depth is pursued (Figure 1). Drainable solids get their deliquoring in a decanter centrifuge mainly when the (under the pool formed) cake is lifted out of the liquor pool and emerges on the dry beach where drainage starts, eventually combined with a displacement washing of the solids [*10*].

Compaction simulation

"Often, simple laboratory studies can provide valuable insight into the physical and chemical mechanisms at work in your process, and extra lab effort can prevent grief during scaleup" [*11*], and this certainly applies also for sludge dewatering in decanter centrifuges.

The author has developed a laboratory protocol that allows the detailed study of sludge centrifugation under controlled laboratory conditions [*12*]. In short, conditioned and pre-thickened sludge is put in a spin tube (Figure 2, right) and centrifuged. Included in the conditioning stage are the addition of cationic polymer (discussed below), as well as the application of a certain vast amount of shearing on the flocculated sludge. Then, after the sludge is centrifuged in the laboratory for a standard 45 min., the water that has squeezed out of the cake (Figure 2, left) is decanted. In the laboratory, it was decided to study the compaction at quasi-equilibrium conditions [*14*] — in the decanter centrifuge, the time required for full cake consolidation is much longer than the cake retention time of 10–15 min.

Next, the compacted sludge cake is removed layer by layer with a laboratory spoon and the dryness of each layer is determined ($\%DS_i$; illustrated at the left side of Figure 3). Together with the recorded mass of each cake segment (w_i) and the cake segment's radial distance from the axis of rotation (R_i), the solids compaction stress (P_S) on an underlying cake layer can, based on the contributions from all the overlying cake layers, approximately be calculated as follows [*12*]:

$$P_S \approx \left(1 - \frac{\rho_{water}}{\rho_{solids}}\right) \times \left(2\pi \frac{N}{60}\right)^2 \times \sum (w_i \times \%DS_i \times R_i) / A \qquad (\mathbf{1})$$

where *N* represents the speed of rotation and *A* is the surface area of the layer (from the spin tube). The compaction curves are obtained then

FIGURE 2. These sludge samples are shown before (right) and after (left) centrifugal compaction in a laboratory spin tube

by plotting, on a $\log_{10}$ scale, cake dryness of the different cake layers versus the respective calculated compressive pressures acting on the center of the cake layers [*3*]. This is illustrated at the right side of Figure 3 for waste sludge that was centrifuged at two speeds of rotation (3,600 or 4,400 rpm) and two amounts used to fill up the tubes (30 or 50 g) to get a thin and thick cake.

Governing parameters

The %DS obtained after centrifugal dewatering of a certain sludge and applied dewatering time is a unique function of P_s which, on its turn, is determined by the rotational speed, the mass and density of the solids. This is exemplified with the data shown in Figure 3. Although clearly different cake dryness data were obtained with a thin and thick cake from the same sludge, with a low and high rotational speed, these data are basically all part of the same compaction curve (characteristic for that particular sludge). When a lower P_s is exerted on the sludge (because a thinner cake is centrifuged, or a lower centrifugal field is applied) the cake dryness is lower and vice versa.

Next to a high *G*-force (high bowl speed, *N*), also a high cake inventory is pursued in a high-solids decanter centrifuge for effective sludge cake dewatering [*3*], since, in that case P_s is increased according to Equation (1). This is achieved by operating the sludge decanter at a so-called "near-plugging" condition, with the circular space between screw conveyor hub and bowl wall almost completely filled up with sludge cake, as illustrated in Figure 1. An extended solids residence time in the machine, and inherently high solids buildup to increase compaction, is made possible by running at a very low differential speed (as low as 1 rpm), as already mentioned.

A higher P_s yields a higher cake dryness. This is true up to a certain P_s (with the value depending on the sludge composition) from whereon further increase of the P_s results only in diminishing to no increase in cake dryness. This distinctive feature of compressible biosolids [*3*] is illustrated in Figure 4, where the compaction curves are shown from three different sludges that mainly differ by their inorganic ($CaCO_3$) fraction. The cake dryness of the best dewatering sludge (upper curve, for sludge with a 70% inorganic fraction) starts to reach a plateau of 40.7%DS only at a high P_s of approximately 160 kPa, whereas a maximal achievable 20.4%DS is already observed at a much lower 40 kPa for the sludge with a 45% inorganic fraction (Figure 4, lower curve). At this compaction stress, the effect of the inorganic fraction is further illustrated: the cake dryness increases from 20.4% to 29.5%DS and 33.9%DS for sludges with 45%, 60% and 70% inorganic fraction, respectively. From this, it is clear that the inorganic solids enmeshed within the sludge floc have a large impact on the cake dryness. An explanation therefore is found in the concept of these solids acting as a skeleton builder [*15*]. When incompressible and drainable solids are present in the compressible and non-drainable biological material, the resulting more-rigid lattice structure allows further dewatering at higher pressures since the compacting cake remains more porous and water can still be expelled out.

Furthermore, next to their presence in the sludge flocs by means of precipitated and enmeshed $CaCO_3$ salts, Ca^{2+} ions can also be present in the sludge flocs as bridging agents between the negatively charged sludge flocs, the latter due to the presence of extracellular polymeric substances (EPS) [*16–17*]. Thanks to their stabilizing effect of the floc structure, the cake dryness increases roughly by 2%DS after centrifugal dewatering at 20 kPa compaction stress when these exchangeable calcium ions are present in the flocs [*18*].

It is widely recognized that wastewater sludge exhibits poor dewaterability due to the presence of EPS, which induce an overall negative surface charge on the flocs. That is why raw waste sludge is first conditioned with cationic polymers to neutralize the sludge's negative surface charge before feeding it to a mechanical dewatering device. Additionally, the polymer conditioning of the sludge flocs also serves the need for more shear-resistant flocs, and the need for the ability to reform flocs after these have been broken down by shear forces, which certainly applies in the case of decanter centrifuges. Indeed, severe mechanical shear occurs during feed acceleration, when the sludge enters the bowl via the stationary feed pipe and then hits

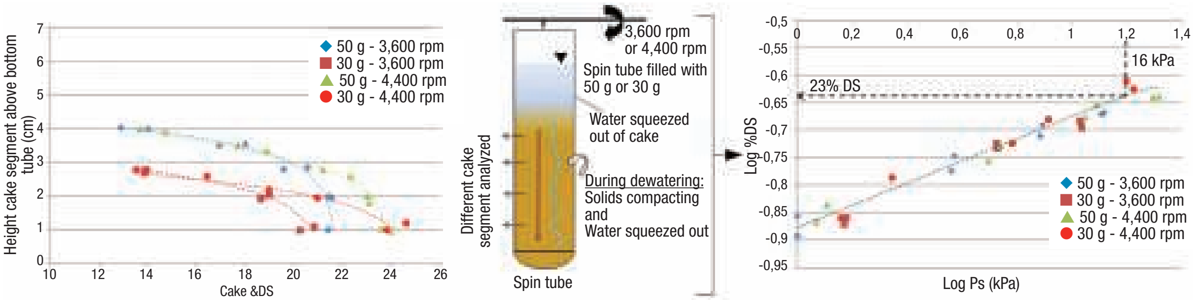

FIGURE 3. Sludge compaction tests at four different centrifugation combinations are shown: two solids amounts were used to fill the spin tubes (30 or 50 g) and two speeds of rotation were applied (4,400 or 3,600 rpm) (DS = dry substance, P_s = solids compaction stress) (adapted from [*13*])

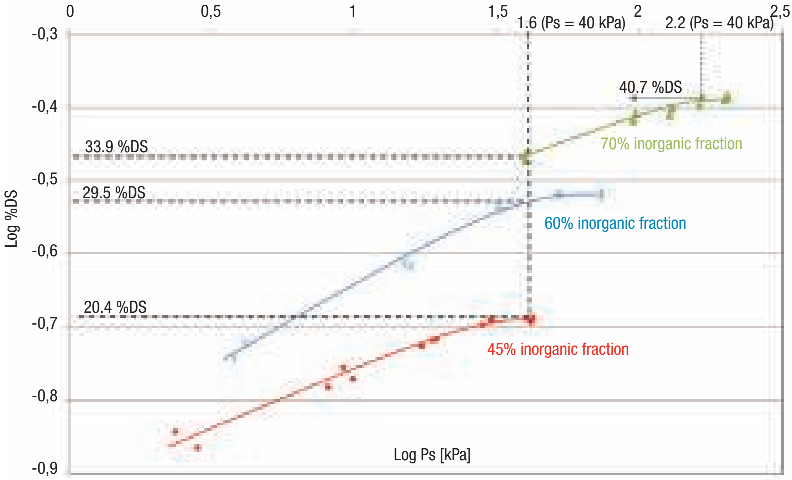

FIGURE 4. Sludges with different levels of inorganic substances can show variable compaction curves

the rotating feeding chamber in the hollow screw conveyor (at very high speed) and, next, the water pool, bringing the new incoming sludge feed suddenly to high centrifugal acceleration [*3*]. For the shear intensive decanter centrifuge applications, non-linear polymers with higher molecular weight and higher degree of structure, with cross-linking between the polymer chains, have been developed. Clearly, the most appropriate polymer (and its optimal dosing) needs to be evaluated for a particular sludge prior to testing it in the field at the full-scale centrifuge (or, at least, the number of commercially available polymers needs to be funneled to a smaller number before going to real-life). This can be accomplished by means of the bench-scale spin tube test described previously. A typical polymer dosing is in the range of 5 to 20 kg polymer active substance per ton of DS.

Prior to the polymer addition, sludge conditioning with polyaluminiumchloride (PACl) can be justified in case the sludge compaction proceeds in the machine to the dryness range where the partially dewatered sludge comes into its sticky phase [*1*]. Without the stickiness-mitigating effect of the PACl, the screw conveyor can experience serious torque issues due to the pasty sludge cake, resulting in the choking of the decanter, seriously hampering its proper working. PACl addition is shown to almost completely eliminate the stickiness of sludge [*19*].

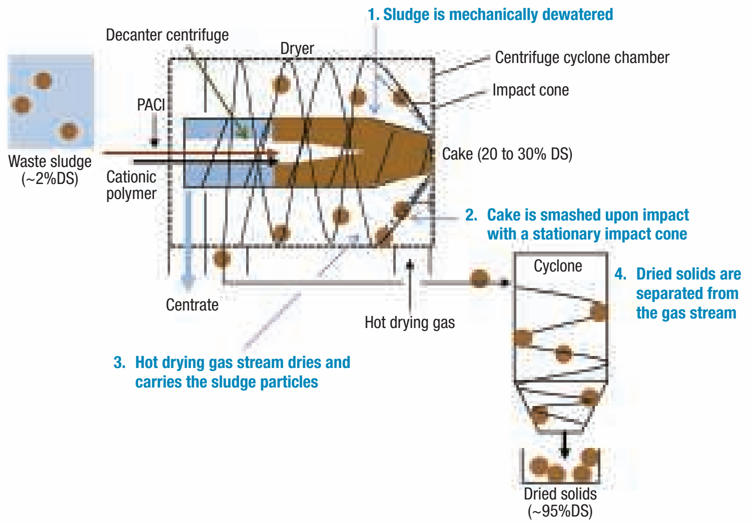

FIGURE 5. A sludge centrifuge and dryer system can be housed in one enclosed machine

Combined system

Figure 5 shows the flow scheme from a centrifuge-dryer system where both unit operations are combined in one compact and enclosed system [*14, 20*]. Briefly, the sludge is dewatered in the decanter centrifuge (as described above), whereupon the discharged cake lumps are immediately disintegrated by impact on a cone surrounding the cake-discharge ports. The resulting solids spray is flash-dried and entrained by a hot gas stream, and a few seconds later, ~95% DS solids are separated in a cyclone.

The huge benefit of this approach is that it has a very simple layout without any need for intermediate storage or conveying of the dewatered sludge cake before feeding it to the next drying stage. In contrast, most industrial sludge-handling installations have a separate dewatering step (a belt filter press or centrifuge, for instance), wherein the resulting cake is then temporarily stored and further conveyed to a standalone sludge dryer. To avoid having the sludge in the dryer equipment go through its sticky phase, the final dried material is recycled and back-mixed with the dewatered cake. This way, the %DS of the blended dryer feed is brought typically beyond 70%DS and from that point, it becomes crumbly and conveyable. In disc, paddle and thin-film dryers, the sticky issues need to be addressed by designing the dryer with high-torque capabilities. The design of the internal rotor is important to overcome the sticky phase in these dryers [*21*].

The continuous transition from centrifugation to flash drying (Figure 5) makes the combined system unique, but it is also its proverbial Achilles' heel. This is because a serious but inherent drawback lies in the fact that the sludge cake leaving the centrifuge is literally never seen, and thus never analyzed. The system resembles a "black box," where liquid sludge is pumped in, and when everything goes well, dry-dust solids come out. Information on the in-between product (cake) is lacking, which makes process optimization difficult. Tests like the previously described spin tube test, to simulate at laboratory scale the compaction occurring at full-scale, can help. ■

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A Primer on Rectangular Tanks

This type of tank is often preferred to cylindrical tanks when space is limited. Follow this guidance to calculate the relevant dimensions and ensure safe construction

Keith Kachelhofer
Hargrove Engineers + Constructors

With the increasing age of many chemical process plants, a challenge often arises when it comes time to find suitable "real estate" to erect and install new storage tanks. Throughout the chemical process industries (CPI), storage tanks are most often thought to be cylindrical tanks with flat bottoms and coned roofs, or leg- or lug-supported cylindrical tanks with dished heads. However, rectangular tanks may provide a viable alternative for facilities that need to store a product under atmospheric pressure but have limited space available to erect a traditional cylindrical tank.

While abundant sources of information are available for cylindrical tanks, engineering guidance related to the specification or design of rectangular tanks is less readily available. For most CPI engineers, the biggest challenge associated with rectangular tanks is how to specify them and how to identify the most economical method of fabrication. As of today, there is no specific published code for rectangular tank design and fabrication. However, the American Petroleum Institute (API) does publish API Bulletin 2V: Design of Flat Plate Structures [*1*], which provides relevant engineering support.

Rectangular tanks are constructred from flat plates that have been welded together with structural beams added to provide rigidity. The plate reinforcement can be implemented using vertical or horizontal stiffeners. This article discusses several detailed design options for the flat plates, stiffeners and stiffened panels that are used to construct rectangular tanks. The structural design of rectangular tanks is primarily an engineering function for mechanical and structural engineers. However, having a fundamental understanding of what is involved with the design process can greatly help chemical engineers and related professionals during the evaluation and specification process. The installation of nozzles (Figure 1) presents a variety of challenges, which are discussed below.

FIGURE 1. This photo shows a reinforced nozzle on a rectangular tank during onsite construction. Note the weep hole located at the bottom of the reinforcement pad

Safety first

Rectangular tanks should always be engineered in the anticipated corroded condition — where the shell plates would be at their anticipated smallest allowable thickness. This will require a corrosion allowance for the wetted surfaces and possibly for the exterior surfaces, as well. If the tank is to be insulated, there might be a concern about moisture becoming trapped under the insulation and causing localized pitting and corrosion on the exterior of the tank.

Analysis of a plate typically starts with a few assumptions: The plate is initially flat, the thickness is small compared to other dimensions, the material is elastic, and deflection does not exceed one half the thickness of the plate. As a general rule of thumb, engineers can make the following assumptions:

- Rectangular tanks with capacities less than 30 ft^3 typically do not require stiffeners
- Tanks with capacities from 30–300 ft^3 in volume will require vertical or horizontal stiffeners
- Tanks above the 300 ft^3 capacity should have vertical and horizontal stiffeners or may have to incorporate stays. Stays are horizontal circular rods (Figure 2) with each end welded to the parallel plates used to form the tank. The stays help keep each plate from experiencing excessive deflection, and the strategic use of stays in the tank design can help to keep overall material costs to a minimum

The governing condition for determining the need for stiffeners will be

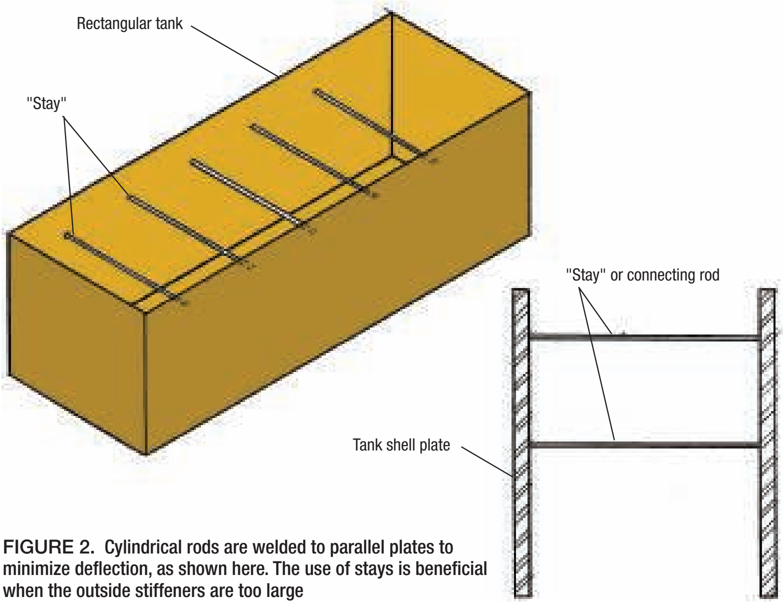

FIGURE 2. Cylindrical rods are welded to parallel plates to minimize deflection, as shown here. The use of stays is beneficial when the outside stiffeners are too large

the height of the tank. As the height of the tank increases, so does the static pressure on the tank. In turn, the size and quantity of structural steel beams required for reinforcement will increase. The key to an economical design is a final tank design that uses the lightest-gage plate material while still providing sufficient reinforcement to ensure needed rigidity.

The size and number of stiffeners depends on the thickness of the tank wall and the spacing of the stiffeners. There is a tradeoff between the plate thickness and the number of stiffeners added to the plate. For instance, there could be a cost savings associated with reducing the plate thickness, but the savings may be offset by the labor required to install plate stiffeners. There are two methods of reinforcing the plate — through the use of vertical or horizontal stiffeners.

The method of horizontal reinforcement is a more direct procedure. Reinforcement incorporating vertical stiffeners requires an assumption of the shell plate thickness and spacing between the stiffeners, which can be an iterative process.

Figure 3 shows a rectangular tank with a top rim stiffener and two vertical stiffeners. Since the plates used to construct the tank are flat, they inherently are not rigid structures when transverse static loads are imposed on the surface. Therefore, structural beams must be attached in order to keep the plates from experiencing excessive deflection. Areas of particular concern are the regions of unsupported plate that exist between the tank corners and between the stiffeners themselves. It is here that the design engineer can combine the properties of the tank wall with those of the selected stiffener (Figure 4) to develop a composite beam, which will have a higher structural rigidity than just the structural member alone. A common method is to consider a section of steel plate having an equal distance between stiffeners.

Determining the spacing between vertical stiffeners is an iterative process. An initial assumption is made for the stiffener spacing followed by a mathematical evaluation of the plate deflection and stress. The anticipated plate deflection and stress are associated with published constants, which are a function of the ratio of the sides of the plate. Depending on how the plate is statically supported, these constants (along with the ratio of the sides) and specifically tailored formulas can be used to determine the plate deflection and bending stress.

If the deflection is excessive (that is, greater than one half of the plate thickness) and stress is greater than the values published in Table 1, then the stiffeners must be spaced more closely together.

Figure 5 shows a rectangular tank with a top rim stiffener and one horizontal (intermediate) stiffener. The advantage of this design is that the intermediate stiffener is located in proximity to where the average load is acting on the surface plane. One stiffener can be attached at the point of loading and act as a beam with a uniform distributed load. The design incorporating horizontal stiffeners is a more direct and simplified approach as opposed to making assumptions with vertical stiffeners.

Tanks incorporating stiffeners can use a wide range of structural shapes, including angle, channel or structural T's. When using a structural angle for reinforcement at the top, the angle can be oriented with the toe out where one face of the angle is oriented against the shell plate. But the intermediate stiffener needs to be attached with the toe facing into the tank wall (one face of the angle oriented outward) to increase structural rigidity, as shown in Figure 6.

Stiffeners are attached to the tank wall by using either continuous fillet welds or stitch welds. For top stiffeners, the inner fillet weld must be continuous (Figure 7) in order to keep the product from becoming trapped

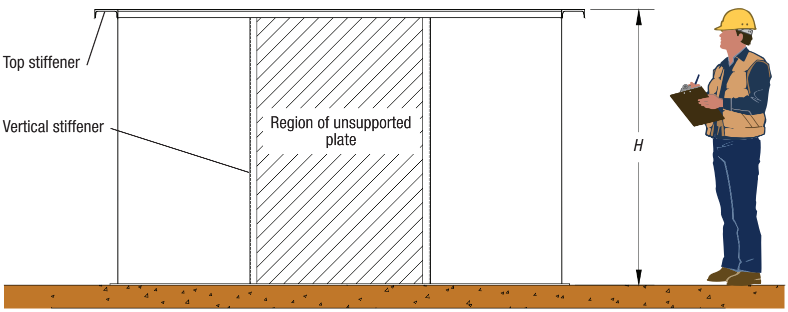

FIGURE 3. Shown here is a rectangular tank with vertical stiffeners. Note the region of unsupported plate between the vertical stiffeners

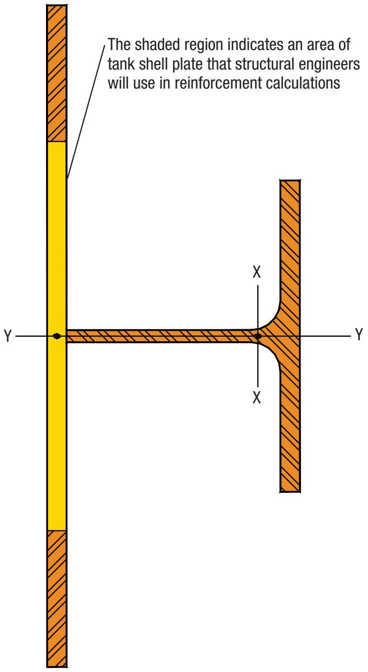

FIGURE 4. **Structural engineers will incorporate a region of the shell plate into the reinforcement calculation to help reduce the size of the reinforcement beam and reduce cost**

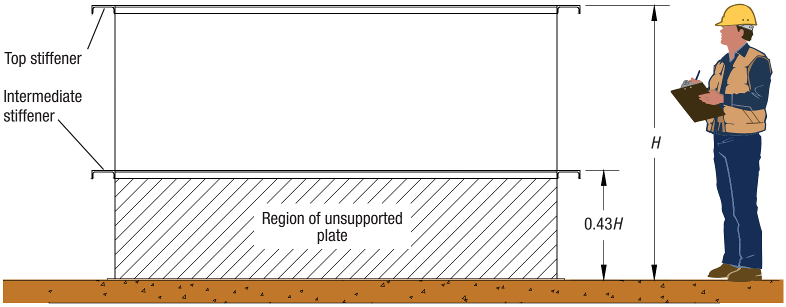

FIGURE 5. **A rectangular tank is shown with a top stiffener and an intermediate stiffener located at 43% of the height of the tank wall, measured from the bottom of the tank**

or seeping between the tank wall and the stiffener. The welds outside of the wetted surface can be stitch welds. Stitch welds (Figure 8) require less labor and use less deposited weld metal. For most applications, a ¼-in. fillet weld is sufficient with weld lengths ranging from 2 to 3 in. and the welds spaced anywhere from 6–10 in. apart.

The corners of the tank walls are regions of high stress due to the discontinuity between the two adjoining plates. Thus, the corner joints will require reinforcement or full penetration welds. Figure 9 shows two corner joint designs that are commonly used during fabrication of rectangular tanks.

A third option is to use a formed corner on the tank shell plate with a full penetration weld, as shown in Figure 10. This is a preferred method for small tanks that use lighter-gauge steel walls (in the range of 10-gauge plate to 1/4-in. plate). The plate is easy to form and the welds are economical.

Adding nozzles to the design

Nozzles installed in rectangular tanks are typically not subjected to high static head pressures, therefore the connection of the nozzle to the tank shell can typically be completed with a fillet weld on the outside of the tank and a fillet weld on the inside of the tank. Typically, there is no need for additional nozzle reinforcement. However, if the nozzle is connected to any rotating equipment, such as pump suction piping, then the tank nozzles should be installed using full penetration welds, followed with a capping fillet weld, as shown in Figure 11.

Since the inherent geometry of the plate does not permit excessive loadings, dynamic equipment should not be suspended directly from the tank nozzles. If mixers and side-wall agitators are to be installed on the tank shell, then a third-party engineering firm should perform finite element analysis on the tank shell and nozzle to ensure adequate reinforcement of the nozzle and shell plate. If unsure as to the need for reinforcement, then the recommendations of API 650, Section 5.7, can be applied [2]. API 650 recommends that openings in tank shells that are larger than an NPS 2 flanged or threaded nozzle be reinforced, as shown in Figure 1. All shell-opening connections that require reinforcement (for example, nozzles, manholes, and cleanout openings) must be attached by welds that fully penetrate the shell, according to API 650.The minimum cross-sectional area of the required reinforcement must not be less than the product of the vertical diameter of the hole cut into the shell and the nominal plate thickness. The cross-sectional area of the reinforcement shall be measured vertically, coincident with the diameter of the opening. Table 5.6(a)(b) in API 650 provides recommendations for reinforcement plate dimensions. Be sure to add a 1/4-in. NPT weep hole in the bottom of the reinforcement pad (Figure 1) to provide the access needed to check for any potential leaks behind the pad.

The bottom plate thickness can be 1/4-in. or 3/8–in. nominal thickness, assuming the plate is entirely supported by a concrete pad. The bottom plate needs to extend beyond the shell plate with sufficient distance to incorporate anchor bolts if desired. Since most rectangular tank geometry is not subjected to high overturning moments, anchor bolts may not be required.

TABLE 1. ALLOWABLE DESIGN STRESS FOR PLATE MATERIAL, PSI

Material Grade	Design Temperature				
	100°F	150°F	200°F	250°F	300°F
SA283 C	15,700	15,700	15,700	15,700	15,700
SA36	16,600	16,600	16,600	16,600	16,600
SA516-70	20,000	20,000	20,000	20,000	20,000
SA240-304	20,000	20,000	20,000	20,000	18,900
SA240-304L	16,700	16,700	16,700	16,700	16,700
SA240-316	20,000	20,000	20,000	20,000	20,000
SA240-316L	16,700	16,700	16,700	16,700	16,700

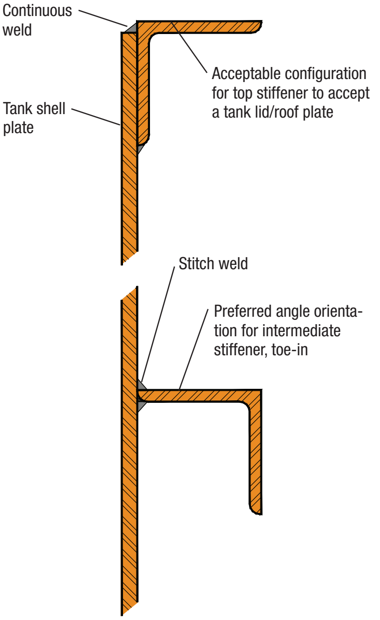

FIGURE 6. Proper orientation of the top stiffener and the intermediate stiffener using angled stiffeners is shown here. The stiffener shown at the top is oriented with the "toe" out, while the intermediate stiffener is oriented with the "toe" in. The intermediate stiffener is stronger with the "toe-in" orientation where a region of the tank plate can be used to resist deflection

If a roof plate is required, then several factors must be considered. Will the roof be permanently attached to the top stiffener or will it be bolted? If the top of the tank is made to be removed, then its weight is very important. Be sure to provide lifting lugs to facilitate maintenance. The size of the tank and the need for access for plant operations and maintenance will determine if the plate needs to be designed for personnel working on the surface.

API 650 recommends a minimum of 45 lb/ft^2 for combined dead load and live loads. This should be sufficient to allow a person to walk on the roof without excessive deflection. For applications that may have hot vapors above the liquid level, it might be prudent to include a corrosion allowance for the inside

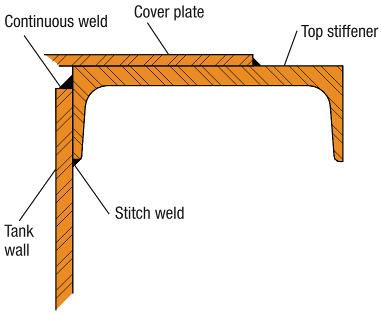

FIGURE 7. This figure shows top stiffener detail with a cover plate. Note the channel is oriented on the *X-X* (strong) axis. See Figure 13 for details

of the plate.

The same design procedures that are used for roof plates are also used for side plates. First, find a ratio of sides where there is a minimal amount of deflection and check the plate stress. Like the vertical stiffeners used for the tank walls, the stiffeners for the roof plate will need to be selected for deflection and stress. For removable roof plates, a general recommendation is to start with 3/16–in. or 1/4-in. plate incorporating channel sections, plate or angle attached, with the *X-X* axis parallel to the roof plates, as shown in Figure 13.

Calculation methodology

There is a simplified method for determining the required shell-plate thickness and the required beam sizes that can be used by CPI engineers. The procedure yields results that are both dependable and conservative. As with most design processes, the first step is to determine the volume of product that needs to be stored. Based upon the available real estate in the plant, the geometry of the tank then needs to be established. Knowing the required volume of the tank, the length of one side can be determined using Equation (1):

$$w = \sqrt[3]{V} \quad (1)$$

FIGURE 8. On this tank, structural reinforcement is provided by a horizontal 'T' shape that is stitch-welded to the side wall of a rectangular stainless steel tank

Where:
w = the length of one side of the tank, ft
V = the required volume for storage, ft^3

The preferred ratio of sides is: 1.5w for the longer side and 0.67w for the shorter side, with the relationships shown in Equations (2) and (3):

$$L_L = 1.5w \quad (2)$$

$$L_S = 0.67w \quad (3)$$

Where:
L_L = the length of the long wall, ft
L_S = the length of the short wall, ft

Next, determine the hydrostatic force on the inside surface of the shell plate. This process involves fundamental statics, where the load imposed on the tank varies linearly with the depth of the fluid (Figure 12).

The pressure at the top of the plate is equal to zero, and the maximum pressure is at the bottom of the tank. To be conservative, the maximum pressure will be used for determining plate deflection and stress using Equation (4):

$$p_{max} = \rho h \quad (4)$$

Where:
ρ = density of the fluid, lb/in.^{3}
h = maximum height of the fluid, in.

The longest wall will govern the design for the tank shell, since this will provide the largest area of the plate that will be subjected to hydrostatic forces. Hydrostatic forces act perpendicular to the surface. The pressure the fluid exerts on the tank walls depends upon its depth. In the case

TABLE 2. MODULUS OF ELASTICITY, E

Material Grade	Design Temperature				
	100°F	200°F	300°F	400°F	500°F
Carbon steel	29.3 x 10^6	28.6 x 10^6	28.1 x 10^6	27.5 x 10^6	27.0 x 10^6
Austenitic stainless	28.3 x 10^6	27.6 x 10^6	27.0 x 10^6	26.5 x 10^6	25.8 x 10^6

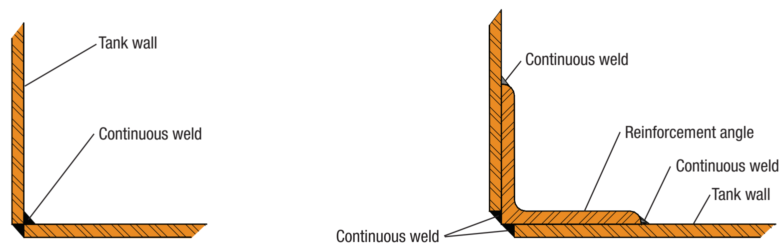

FIGURE 9. Two corner joint designs are shown here. The preferred method is the use of a reinforcement angle with continuous fillet welds

of water, there is 1 lb of pressure for every 2.31 ft of depth. The force on the shell plate can be calculated using Equation (5):

$$F_r = \frac{1}{2}\left(0.036\frac{lb}{in.^3}\right)\left(h^2\right)(S.G.) \quad (5)$$

Where:
h = the maximum height of the fluid level, in.
F_r = the resultant force, lb/in.
S.G. = the specific gravity of the fluid stored (that is, the ratio of a specific fluid density to the density of water), dimensionless

The use of a horizontal intermediate stiffener located between the top stiffener and the bottom of the tank is the simplest approach. It is recommended that the intermediate stiffener be located at 0.43*H* from the bottom of the tank [*7*] where *H* is the height of the tank wall (Figure 5).

The next step is to determine the required plate thickness, using Equation (6):

$$t_{reqd} = 0.3(h)\sqrt{\frac{0.036(S.G.)(h)}{\sigma_a}} \quad (6)$$

Where:
S.G. = the specific gravity of the fluid stored, dimensionless
σ = the allowable bending stress for the steel plate, psi
h = the maximum height of the fluid level, in.

Table 1 provides a list of common steel alloys and their respective bending stresses. Table 2 provides the modulus of elasticity of various grades of steel at various temperatures. The modulus of elasticity is a number that measures an object's resistance to being deformed elastically when a force is applied.

If the tank is in a corrosive environment, then an appropriate corrosion allowance needs to be added to the required plate thickness. The most common corrosion allowances range from 1/16 to 3/16 in.

The next step is to determine the required moment of inertia for the stiffener located at the top of the tank (this is shown in Figure 5). Inertia is a measure of a body's ability to resist movement, bending or rotation. The moment of inertia is a measure of a beam's stiffness in relation to its cross-section. As a beam's moment of inertia increases, its ability to bend decreases. For flat plate, channel, structural *T* and angle, the highest moment of inertia is located on the *X-X* axis as shown in Figure 13. The minimum required moment of inertia for the top stiffener beam can be found using Equation (7):

$$I_{top} = \frac{(0.06F_r)\left(L_L^{\,4}\right)}{192(E)\left(t_{nom}\right)} \quad (7)$$

Where:
L_L = length of the longest tank wall, in.
E = modulus of elasticity, psi
t_{nom} = the nominal thickness of plate to be used, in.

The minimum required moment of inertia for the intermediate stiffener located between the top stiffener and the tank bottom can be determined using Equation (8):

$$I_{int} = \frac{(0.3F_r)\left(L_L^{\,4}\right)}{192(E)\left(t_{nom}\right)} \quad (8)$$

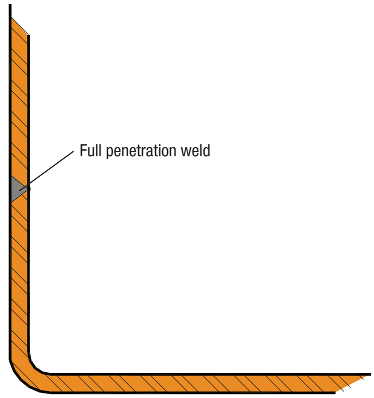

FIGURE 10. The preferred corner joint for light-gauge plate ranging from 10GA to 1/4-in. nominal thickness. Forming the corners and providing one full penetration weld helps to strengthen the corner and minimize plate distortions during welding

Be sure to convert your wall length from feet to inches when using Equations (7) and (8) in order to maintain consistent units. The top and intermediate stiffeners can be any variety of structural shapes, such as channels, T-shapes or angles. Figure 13 shows examples of common structural shapes that can be used for stiffeners. For smaller tanks, structural angles are the most economical option, and they provide a surface onto which a cover plate or lid can be bolted or welded. Larger tanks will typically incorporate larger structural members, such as channels. The structural member must have a moment of inertia that is greater than what was calculated above for the top and intermediate stiffeners. The mechanical properties of structural members can be found online at the Engineers Edge website [*8*].

When selecting a structural member from the tables online provided in Ref. 8, it is imperative to select a moment of inertia on the *X-X* axis of the beam, as shown in Figure 13. This is typically denoted as I_{XX} where *I* is the moment of inertia of the shape and the subscript *X-X* represents the moment of inertia about the *X-X* axis. When possible, try to use the same structural member for both the top

TABLE 3. PLATE CONSTANTS FOR RECTANGULAR PLATES WITH ALL EDGES FIXED AND UNIFORM DISTRIBUTED LOAD

W/H	1.00	1.20	1.40	1.60	1.80	2.00	Infinity
β	0.1386	0.1794	0.2094	0.2286	0.2406	0.2472	0.2500
α	0.0138	0.0188	0.0226	0.0251	0.0267	0.0277	0.0284

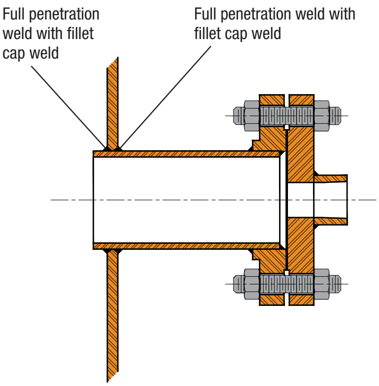

FIGURE 11. This figure shows details of a nozzle that is attached to a tank using full penetration welds and a capping fillet weld on the inside and outside of the nozzle

stiffener and the intermediate stiffener to help minimize cost.

Plate deflection and stress

Once the stiffeners have been sized, it is good practice to check the plate deflection and plate stress. The first step in calculating plate deflection and stress is to determine the length of the sides supported by adjacent plates or structural members. Figure 14 shows a rectangular plate with dimension *W* being the width of the plate and dimension *H* being the height of the plate. Next determine the ratio of sides using Equation (9):

$$R_{plate}=\frac{W}{H} \qquad (9)$$

Where:
W = width of unsupported plate, in.
H = height of unsupported plate, in.

The width of unsupported plate is dependent on the location between stiffeners. When calculating the worst case scenario for horizontal tanks, the longest tank shell plate (L_L) can be used in place of *W* as indicated in the example problem. The ratio of plate width to plate height is required to find the appropriate constants in Table 3. If a calculated ratio is between two published ratios in the table then interpolation is permitted. Calculated ratios above 2.0 will default to the infinity values published in Table 3 [4].

The maximum hydrostatic pressure is determined by Equation (4). The plate deflection is determined by Equation (10):

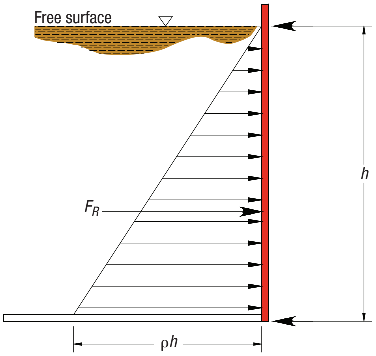

FIGURE 12. This diagram shows the hydrostatic forces on the tank wall. The maxium height of the fluid level is denoted as '*h*' and is recommended to be 6 in. below the top of the tank wall

$$\Delta=\frac{\alpha pH^4}{Et^3} \qquad (10)$$

Where:
α = constant from Table 3
p = hydrostatic pressure, psi
H = height of the tank shell plate, in.
E = modulus of elasticity, psi (from Table 2)
t = nominal thickness of the tank shell plate, in.

The plate deflection should be less than half the thickness of the plate being used on the tank. The plate stress is determined using Equation (11):

$$\sigma=\frac{\beta pH^2}{t^2} \qquad (11)$$

Where:

β = constant from Table 3

Sample problem

A chemical plant requires a tank with 250-ft^3 storage capacity and an open top. Assume the tank bottom is resting on a concrete pad with the bottom fabricated from 1/4-in. plate. The fluid is water at ambient conditions.

The objective is to provide an engineering design for the required shell plate thickness and the required stiffeners. The tank dimensions are determined using Equation (1), as shown in Equation (12):

$$w=\sqrt[3]{250\,ft^3}=6.29\,ft\approx 75.5\,in. \qquad (12)$$

The length of the long side of the tank is determined using Equation

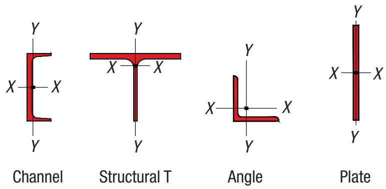

FIGURE 13. Common structural shapes used for reinforcement are shown here. The *X-X* axis is considered to be the strongest axis to resist movement under loading. It is important to attach the reinforcement beam to the tank shell with the *X-X* axis parallel to the plate

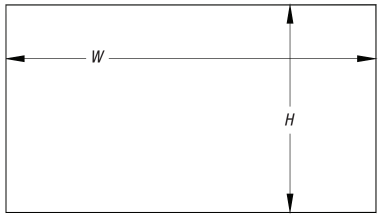

FIGURE 14. Dimensions of unsupported plate located between the stiffeners or adjacent plate

(2), as shown in Equation (13):

$$L_L=1.5(6.29\,ft)=9.5\,ft\approx 114\,in. \qquad (13)$$

The length of the short side of the tank is determined using Equation (3), as shown in Equation (14):

$$L_S=0.67(6.29\,ft)=4.2\,ft\approx 50\,in. \qquad (14)$$

Since we know the tank sides are 9.5 ft-by-4.2 ft then the floor area is 40 ft^2. Dividing the required volume of 250 ft^3 by the area of 40 ft^2 the required tank height is 6.25 ft (75 in.)

The reaction force on the plate is calculated using Equation (5), as shown in Equation (15):

$$F_r=\frac{1}{2}\left(0.036\frac{lb}{in.^3}\right)(75^2)(1) \qquad (15)$$

$$=101.25\frac{lb}{in.}$$

Next, the required minimum plate thickness is calculated using Equation (6), as shown here in Equation (16):

$$t_{reqd}=0.3(75in.)\sqrt{\frac{0.036(1)(75in.)}{16{,}600\,psi}}=0.286in.\approx\frac{5}{16}in. \qquad (16)$$

Since the required thickness is

above 1/4-in. then assume 5/16-in. plate. The required moment of inertia for the top stiffener beam is calculated using Equation (7), as shown in Equation (17):

$$I_{top} = \frac{(0.06)\left(101.25\frac{lb}{in.}\right)(114in.)^4}{192(2.93x10^6\,psi)(0.3125in.)} = 0.583in.^4 \quad (17)$$

Use an angle 2.5 in. × 2.5 in. × 0.25 in., with a moment of inertia I_{xx}= 0.703 in.^{4}

Next, calculate the required moment of inertia of the intermediate stiffener using Equation (8), as shown below in Equation (18):

$$I_{int} = \frac{(0.3)\left(101.25\frac{lb}{in.}\right)(114in.)^4}{192(2.93x10^6\,psi)(0.3125in.)} = 2.918in.^4 \quad (18)$$

Use an angle 4 in. × 4 in. × 1/4 in. with a moment of inertia I_{xx} = 3.04 in.^{4} or use a C4 × 5.4 channel (this is a designation of the American Inst. of Steel Construction; Details can be found in [6]), with a moment of inertia I_{xx} = 3.85 in.^{4}

Check the plate deflection below the intermediate stiffener. This area of plate will experience the highest pressure acting on the plate. Determine the pressure using Equation (4), as shown in Equation (19):

$$p = \left(0.036\frac{lb}{in^3}\right)(72in.) = 2.59\,psi \quad (19)$$

Note: the fluid height is assumed to be 72 in.

The ratio of plate dimensions is determined using Equation (9), as shown in Equation (20):

$$R_{plate} = \frac{W}{0.43H} = \frac{9.5\,ft}{2.68\,ft} = 3.53 \quad (20)$$

Remember that the recommended location of the intermediate stiffener [7] is 0.43*H,* which is 2.68 ft (32.25 in.) from the bottom of the tank.

The plate deflection is determined using Equation (10), as shown below in Equation (21):

$$\Delta = \frac{(0.0284)(2.59\,psi)(32.25\,in)^4}{(2.93x10^6\,psi)(0.3125in.)^3} = 0.088\,in. \quad (21)$$

The plate deflection is less than 0.156 in. so the plate thickness is determined to be sufficient. Finally, the plate stress can be determined from Equation (11), as shown in Equation (22):

$$\sigma = \frac{(0.25)(2.59\,psi)(32.25\,in)^4}{(0.3125in.)^2} = 6{,}896\,psi \quad (22)$$

The plate stress is below the published plate stress in Table 1 so the tank design can be considered to be safe. ■

Edited by Suzanne Shelley

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Increase Profits in Size-Reduction Plants

Material- and energy-balance models can help to identify potential opportunities

Jimmy Kumana
Kumana & Associates

The processing of mined minerals in aboveground facilities relies on several energy-intensive unit operations, such as comminution, screening and material transport. Crushing operations typically produce a wide distribution of product rock sizes. This article demonstrates that selecting a crusher that is able to produce a narrower size-distribution profile can not only reduce power consumption (by minimizing the need for recycle), but can substantially increase revenue and profit margins as well, by maximizing the production of the higher-value fractions. The same strategy can also be applied to solids-processing operations in other process industries, such as catalyst manufacturing, cement manufacturing, coal-based chemicals production, production of rock phosphate, and for steam-generation systems that use lump coal as the fuel.

Material- and energy-balance models provide a simple and effective tool to identify and quantify such opportunities. An illustrative example is provided below.

Kumana & Associates

The ability to obtain sharper product-distribution profiles during size-reduction efforts can help to significantly improve the profit margin of the facility

Watching energy use

Historically, the production of a mineral commodity has been perceived as two distinct stages: 1) mining to extract the commodity from the ground; and 2) processing the commodity to convert it into a marketable end product. The example used in this article is based on the author's experience in quick-lime production plants, but the lessons learned are generically applicable to all unit operations that involve size reduction.

The mining and minerals industry is a major energy consumer — fuels are required for excavation and rock-moving equipment, while electric power is consumed during rock and ore processing for size reduction (crushing and grinding), size grading (screening), conveying (material transport) and other operations (Figure 1).

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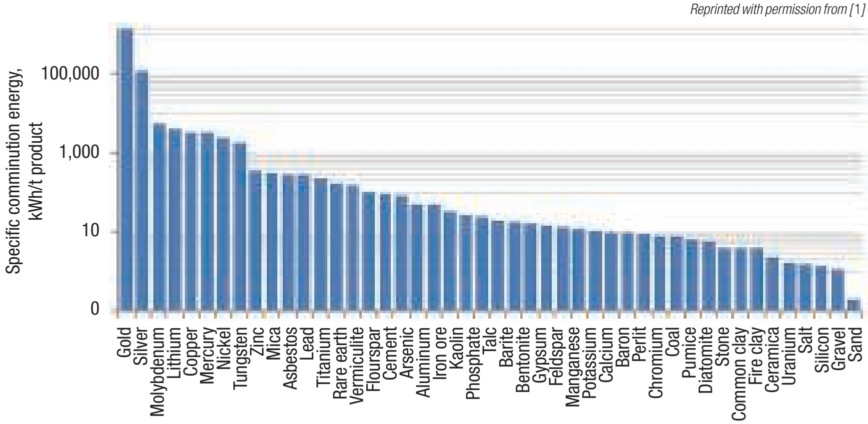

FIGURE 1. The average specific energy required for the size reduction varies by the mineral type

On the basis of production volume, coal and limestone aggregate production are the two dominant processes that employ these energy-intensive unit operations to process the commodities once they are mined. Today, three-quarters of the crushed stone production in the U.S. is limestone and dolomite, followed by, in descending order of tonnage: granite, traprock, sandstone and quartzite, miscellaneous stone, marble, slate, calcareous marl, shell, volcanic cinder and scoria.

Limestone is found in sedimentary deposits, and is composed mostly of the mineral calcite; it comprises about 15% of the earth's sedimentary crust. Limestone is a widely used building block in the construction industry and is the principal raw material from which aggregate, cement, lime and building stone are made. For the purposes of this article, limestone will be used as a representative of all crushed rock.

The aggregates industry serves as an excellent example of how these three main unit operations — size reduction, screening and conveying — can be optimized on an integrated basis. The focus should be on modeling the aggregate production process as a whole, rather than on individual crushing, grinding, or conveying operations.

Process description

Rocky minerals are mined using explosives and quarrying. Large boulders are typically transported by a font-end loader to primary jaw crushers that break the rock into 6–8-in. stones. Further size reduction is done in multiple stages using gyratory crushers, impact crushers and cone crushers, with intermediate transport usually via a conveyor belt.

After crushing, the rock material or final product (as in the case of lime and cement) may be further reduced in size in grinding mills. There are a variety of different grinding mills, including rod and ball mills, semiautogenous (SAG) mills and autogenous mills. In the grinding mills, the impact of the minerals grinding against each other and against the steel balls in the mill breaks down the size of the minerals even further. The milling process also includes vibrating screens that sort the crushed material into the desired size fractions.

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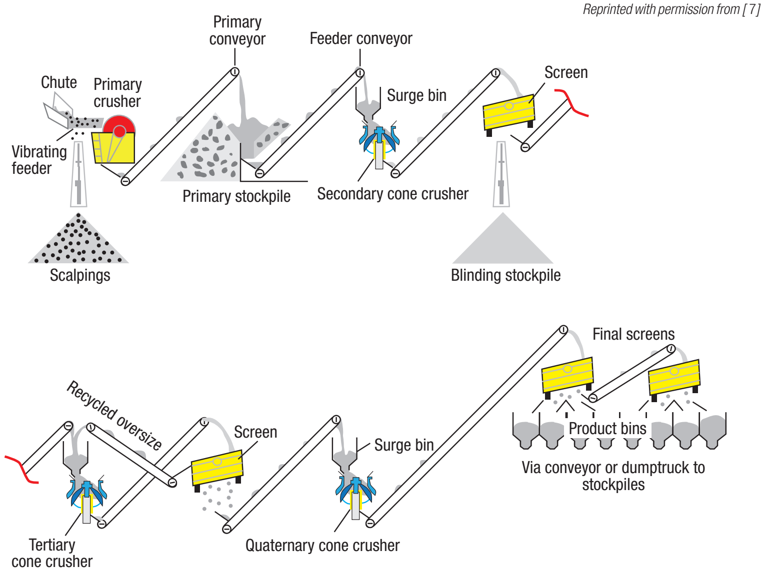

FIGURE 2. The flowsheet shows the major operations that are used in a typical quarrying operation

A review of basic statistics

The probability distribution function (PDF) of crushed rock size tends to follow a "normal" (also called Gaussian) distribution, which is described by Equation (1):

$$f(x) = \frac{1}{\sigma\sqrt{2\pi}} \exp\left[(x - \quad)^2 / 2\sigma^2\right] \quad (1)$$

Where:

μ = the mean or expected value (and also the median and mode) of parameter *x*

σ = the standard deviation of *x* (defined as the square root of the variance σ^2)

$t = (x - \mu)/\sigma$

If μ = 0 and σ = 1, then the distribution is called the standard normal distribution.

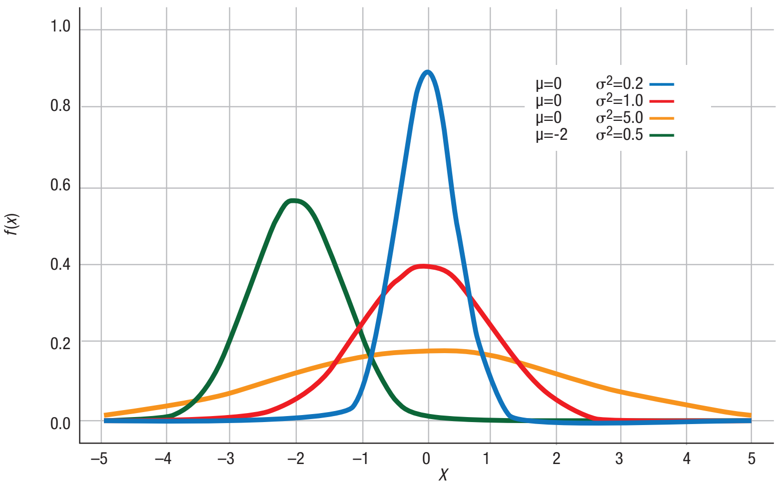

FIGURE 3. Shown here is the frequency distribution function for parameter *X*, under various combinations of mean and variance

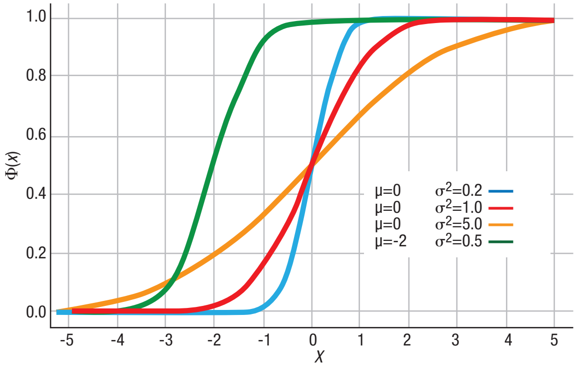

FIGURE 4. This figure shows the cumulative distribution function (CDF) for parameter *X*, under the same conditions were used as in Figure 3

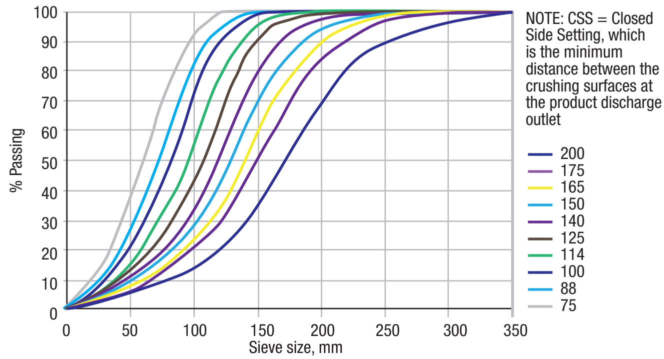

FIGURE 5. Shown here is an example of cumulative product size-distribution data for various models of jaw crushers from the same manufacturer (in this case, Terex; www.terex.com)

Graphical representations of the parameter *x* with various types of normal distributions are shown in Figure 3. The red curve is a standard normal distribution. If the variance is reduced, a narrower distribution with a higher peak is obtained, as shown by the blue curve. If the mean μ has a non-zero value, then the curve shifts to the left or right, as shown by the green curve.

The cumulative distribution function (CDF) of the normal distribution, usually denoted with the capital Greek letter Φ, is the integral, as shown in Equation 2:

$$\Phi(x) = \frac{1}{\sqrt{2\pi}} \int_{-\infty}^{x} e^{-t^2/2} dt \qquad (2)$$

It should be noted that Equation (2)is not integrable explicitly in terms of algebraic functions, and must be solved numerically using published tables.

Figure 4 shows the cumulative particle-size distribution of mixtures with varying values of μ and σ. Figure 3 shows the actual frequency distribution for the same mixtures. Both figures actually convey the same information, but in different formats.

In reality, size distribution will not be quite Gaussian, because the minimum size cannot be negative, and real data seldom follow the smooth lines shown in the idealized figures above. In large part, the discrepancy is due to the shape of the size-reduced rocks — because it is impossible to represent the size of non-spherical particles with a single dimensional value. Nevertheless, the method described here provides a sufficiently good approximation for practical purposes, and certainly helps the engineer to think clearly about the design issues at hand.

Most reputable manufacturers will publish the cumulative product size distribution that is achievable for various models of their crushers (Figure 5 shows an example), as an aid to design engineers seeking to select the most suitable model.

An illustrative example

The simple generic size-reduction circuit shown in Figure 6 demonstrates certain design concepts and principles in a generalized way, and should not be construed as being representative of any particular existing plant.

In this example, a simple material-balance model was developed to estimate the benefits of choosing a crusher that produces a sharper size distribution (that is, with a lower standard deviation) of crushed limestone rock product sizes (Figures 7 and 8). The existing crusher is designated Design A and the proposed new crusher is designated Design B.

Power was correlated versus feed capacity using data from the brochure of a jaw crusher manufacturer [2] for nominal 8-in. feed (defined as 80% of mass being less than 8 in. in dia.). The correlation is shown in Equation (3):

$$Consumed\ kW = 0.00047(tons/h)^2 + 0.00263(tons/h) - 0.073 \qquad (3)$$

Conveying power is a function of many parameters, the most significant being load, length, conveyor layout (that is, the number of changes in elevation and direction), and the incline angle. The calculation is rather complex [3–5] but for any specific layout, the power consumption can be expressed in simplified form using Equation (4):

$$kW = 0.75\,HP = (A + mW)L \qquad (4)$$

Where:
A and *m* = machine- and layout-dependent dimensional constants
W = the mass of material being transported, ton/h
L = the length of the conveyor, ft (assumed to be 400 ft and 500 ft, respectively, for the conveyors 1 and 2 represented in Figure 6).

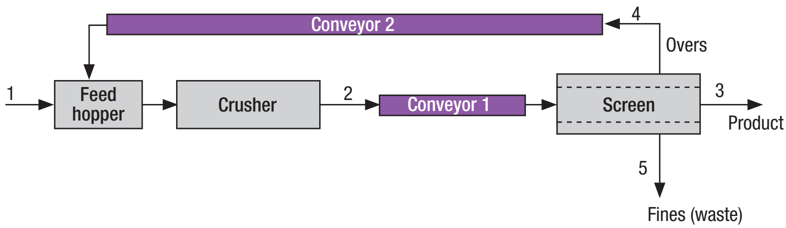

FIGURE 6. This conceptual flow diagram shows the typical steps involved in a size-reduction (comminution) process

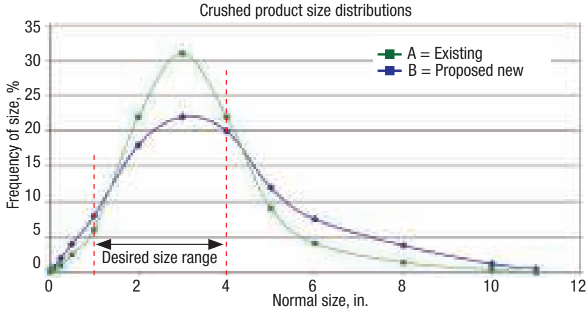

FIGURE 7. Comparing the frequency size distributions of product from crusher designs A and B clearly shows that Design B, with the same mean but lower variance, will have more material (the area under the curve) in the desired size range compared to Design A

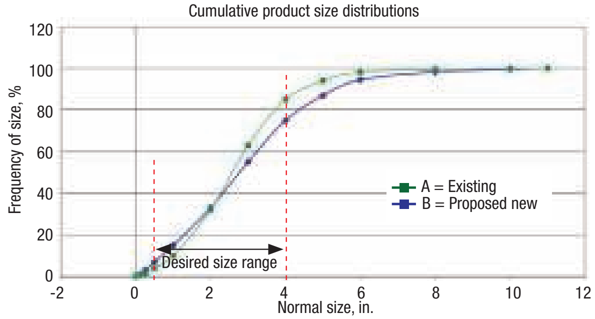

FIGURE 8. This figure uses the same data as Figure 7, but plotted as cumulative weight percent on the Y-axis. It also shows that the fraction of product in the desired size range (from 1 to 4 in. dia) is 84-10 = 74% of the feed for Design B, versus 76-15 = 61% for Design A

Strictly speaking, the constants A and m should be different for each section of the conveyor, but for simplicity we will assume that they are the same, with A = 0.0375, and m = 0.0625 when W is in tons/h and L is in ft.

Now, the material balance can be calculated (some iteration may be required for the recycle rate of overs), as can the power consumption for the two cases.

Tables 2 and 3 show the material balances for designs A and B, for the flowsheet of Figure 6. The crusher and conveyor power consumption associated with each stream is also shown.

It can be seen from the comparison in Table 4 that while the energy intensity of Design B is lower by 22%, the primary financial benefit actually derives from higher sales revenue that results when production is shifted from the lower-value fraction (fines) to the higher-value middle fraction of final crushed product. The simulation model also reveals that it is much more economic to recycle the "overs" fraction for making more high-value middle fraction than to sell it as a low-value product on the market.

For the assumed ex-factory product prices and power costs (that is, exclusing transportation and distribution costs and middleman profits), the profit-improvement potential from employing a crusher with a sharper probability-distribution curve is significant (8%), which could help to justify the capital cost associated with replacing the existing unit. Of course, if the site has different product prices, the potential increased profit could be higher or lower.

Due to the particular design of this circuit — despite the 22% energy-savings potential — the overall impact of this upgrade on plant operating costs is minimal. However, if the conveyor lengths were longer and the inclines greater, then the reduction on power costs could have had a more significant impact on the bottom line.

Although this example uses an admittedly oversimplified model, it does provide important insights into the key drivers of economics in plants where size-reduction operations are a dominant portion of operating costs. Actual design and optimization should be carried out

TABLE 1. CRUSHER PRODUCT SIZE DISTRIBUTION DATA FOR THE ILLUSTRATIVE EXAMPLE

Assumptions					Size	Value
Capacity	150	ton/h			range, in.	$/ton
Feed rock size	8	in.		fines	< 1	4
Power cost	9	cents/kWh		middle	1–4	20
Operating time	4,200	h/yr		overs	>4	9

TABLE 2. MATERIAL BALANCE AND POWER CONSUMPTION FOR DESIGN A

	Crusher Design A				
Stream number	**1**	**2**	**3**	**4**	**5**
Bulk (>12 in.)	135	0	0	0	0
Overs (>4 in.)	14	50.9	0	50.9	0
Product (1–4 in.)	2	122.2	120.0	2.1	0
Fines (< 1 in.)	0	30.5	1.2	0.5	28.8
Total, ton/h	150	203.6	121.2	53.6	28.8
			Trial recycle	53.6	
Size distribution, assumed					
Bulk (>12 in.)	90%	0%	0%	0%	0%
Overs (>4 in.)	9%	25%	0%	95%	0%
Product (1–4 in.)	1%	60%	99%	4%	0%
Fines (< 1 in.)	0%	15%	1%	1%	100%
	100%	100%	100%	100%	100%
Power consumption					
Crusher, HP		26.6		0	
Conveyor, HP		12.7		3.7	
Total power, kW		29.5		2.8	
Power cost, $/h		2.7		0.2	
$1,000/yr		11.1		1.0	

TABLE 3. MATERIAL BALANCE AND POWER CONSUMPTION FOR DESIGN B					
Stream number	1	2	3	4	5
Bulk (>12 in.)	135	0	0	0	0
Overs (>4 in.)	14	26.7	0	26.7	0
Product (1–4 in.)	2	133.6	132.5	1.1	0
Fines (< 1in.)	0	17.8	1.3	0.3	16.2
Total, ton/h	150	178.1	133.8	28.1	16.2
			Trial recycle	28.1	
Size distribution, assumed					
Bulk (>12 in.)	90%	0	0%	0%	0%
Overs (>4 in.)	9%	15%	0%	95%	0%
Product (1–4 in.)	1%	75%	99%	4%	0%
Fines (< 1 in.)	0%	10%	1%	1%	100%
	100%	100%	100%	100%	100%
Power consumption					
Crusher, HP		20.4		0	
Conveyor, HP		11.1		1.9	
Total power, kW		23.7		1.4	
Power cost, $/h		2.1		0.1	
$1,000/yr		8.9		0.5	

using more rigorous simulations, as described in Ref. 6.

Closing thoughts

The illustrative example provided in this article highlights four important truisms:

1. The ability to obtain sharper product-distribution profiles in size-reduction operations can significantly improve the profit margins of the facility
2. Even simplified material balance models are an effective tool to help engineers gain insight into areas of potential improvement, and to assess the economic impact of capital improvements
3. While energy savings for crushing and grinding circuits may be high in relative percentage terms, the value of optimizing the product mix has a far greater positive impact on overall plant economics
4. For new plant designs, the method described here can help the engineer to select the optimum crusher and screen combination for various, alternative plant-ayout options

Cost-effective energy conservation should be a performance goal for all manufacturing operations. Historically, most companies turn their attention to economic optimization only after the product has become "mature" and has become a commodity — when it becomes important to protect profit margins in the face of price competition. Unfortunately, by then it is often too late, because competitors may have not only achieved parity, but may have even gained some technological edge through innovation. By ignoring the opportunity to optimize the process design, complacent companies leave open a gaping hole for their rivals to acquire a competitive edge through the development of a more energy-efficient process with lower costs. ■

Edited by Suzanne Shelley

TABLE 4. COMPARISON OF ECONOMICS FOR CRUSHER DESIGNS A AND B					
	Design A		Design B		Difference %$
	ton/h	$/h	ton/h	$/h	
Main product	121.2	2,425	133.8	2,676	10.4
Fines	28.8	115	16.2	65	-43.8
Total flow	150	2,540	150	2,741	7.9
	kW	$/h	kW	$/h	D, %
Crusher power	29.5	2.7	23.7	2.1	-19.8
Conveyor power	2.8	0.2	1.4	0.1	-47.5
Total	32.3	2.9	25.1	2.3	-22.2
Intensity, kWh/ton	0.215	n/a	0.167	n/a	-22.2
Marginal profit, $/h		2,536.8		2,738.3	7.9

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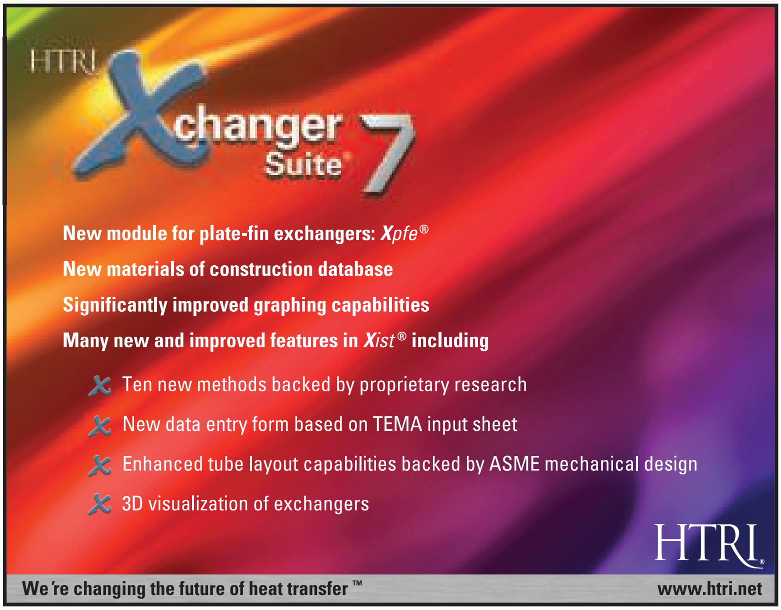

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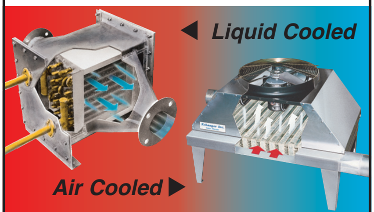

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10	25	40	55	70	85	100	115	130	145	160	175	190	205	220	235	250	265	280	295	310	325	340	355	370	385	400	415	430	445	460	475	490	505	520	535	550	565	580	595
11	26	41	56	71	86	101	116	131	146	161	176	191	206	221	236	251	266	281	296	311	326	341	356	371	386	401	416	431	446	461	476	491	506	521	536	551	566	581	596
12	27	42	57	72	87	102	117	132	147	162	177	192	207	222	237	252	267	282	297	312	327	342	357	372	387	402	417	432	447	462	477	492	507	522	537	552	567	582	597
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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Economic Indicators

2013 2014 2015

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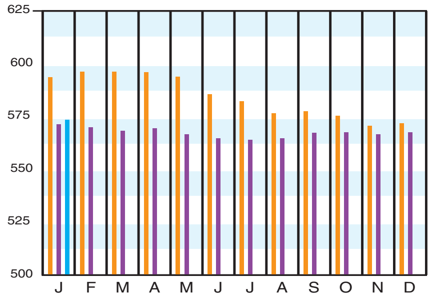
CHEMICAL ENGINEERING PLANT COST INDEX (CEPCI)

(1957-59 = 100)	Jan. '15 Prelim.	Dec. '14 Final	Jan. '14 Final
CE Index	573.4	575.7	572.8
Equipment	694.9	698.8	695.6
Heat exchangers & tanks	636.5	642.5	632.6
Process machinery	663.5	662.8	657.5
Pipe, valves & fittings	868.9	872.2	883.5
Process instruments	407.3	410.7	412.7
Pumps & compressors	948.7	943.4	928.7
Electrical equipment	513.9	515.2	515.9
Structural supports & misc	758.0	765.8	762.3
Construction labor	322.2	322.1	318.1
Buildings	547.2	546.4	537.7
Engineering & supervision	321.2	319.1	323.1

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

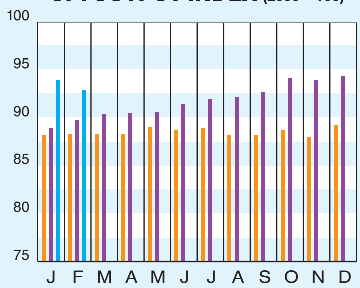
Annual Index:
2007 = 525.4
2008 = 575.4
2009 = 521.9
2010 = 550.8
2011 = 585.7
2012 = 584.6
2013 = 567.3
2014 = 576.1



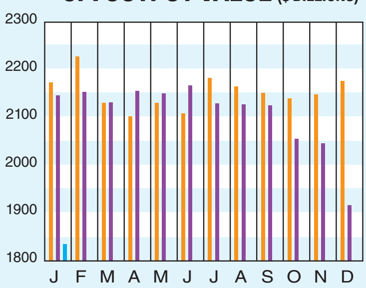
CURRENT BUSINESS INDICATORS

	LATEST	PREVIOUS		YEAR AGO
CPI output index (2000 = 100)	Feb.'15 = 92.9	Jan.'15 = 92.9	Dec.'14 = 93.5	Feb.'14 = 89.3
CPI value of output, $ billions	Jan.'15 = 1,835.6	Dec.'14 = 1,924.3	Nov.'14 = 2,024.2	Jan.'14 = 2,146.8
CPI operating rate, %	Feb.'15 = 77.7	Jan.'15 = 77.7	Dec.'14 = 78.3	Feb.'14 = 75.4
Producer prices, industrial chemicals (1982 = 100)	Feb.'15 = 241.8	Jan.'15 = 246.4	Dec.'14 = 271.0	Feb.'14 = 298.6
Industrial Production in Manufacturing (2002=100)*	Feb.'15 = 101.3	Jan.'15 = 101.5	Dec.'14 = 101.8	Feb.'14 = 98.0
Hourly earnings index, chemical & allied products (1992 = 100)	Feb.'15 = 156.6	Jan.'15 = 158.3	Dec.'14 = 157.3	Feb.'14 = 157.1
Productivity index, chemicals & allied products (1992 = 100)	Feb.'15 = 107.0	Jan.'15 = 108.2	Dec.'14 = 107.2	Feb.'14 = 106.6

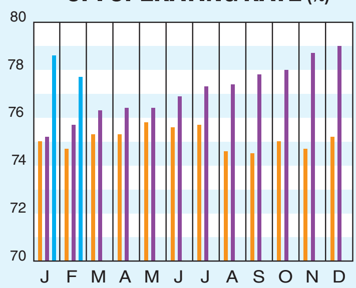
CPI OUTPUT INDEX (2000 = 100)



CPI OUTPUT VALUE ($ BILLIONS)



CPI OPERATING RATE (%)



*Due to discontinuance, the Index of Industrial Activity has been replaced by the Industrial Production in Manufacturing index from the U.S. Federal Reserve Board.
Current business indicators provided by Global Insight, Inc., Lexington, Mass.

CURRENT TRENDS

The preliminary value for the January 2015 *CE* Plant Cost Index (CEPCI; top; most recent available) edged downward from the previous month's value for the fourth consecutive month. The first calculated value for 2015 sits only 0.11% higher than the year-earlier value, the smallest gap between current and year-earlier values in more than a year. Also, the annual average for 2014 was calculated at 576.1. This total is higher than the annual value for the previous year (567.3), but still below the 2012 annual average (584.6).

Circle 21 on p. 78 or go to adlinks.chemengonline.com/56196-21

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