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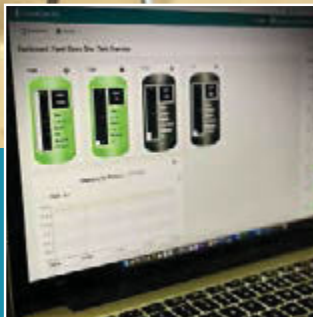
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COVER STORY

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Folio Editorial Excellence Award Winner

Ida Teaches a Tough Lesson

Think carefully about where you keep irreplaceable items

HURRICANE IDA devastated Louisiana in late August. Then, despite being downgraded to a tropical storm, Ida wreaked havoc and destruction when it hit the Northeastern United States in early September — as I personally can attest. Over 10 inches of storm water flooded the basement of the house in New York City my family has lived in since 1978.

Torrential rain from the storm had started but the basement was dry when I checked. Then, near midnight, I got a frantic call from a neighbor. “Our basement is full of water,” he screamed. Scurrying downstairs, I discovered a flood of storm water that reached above the first step of the stairs. By morning, the water had receded, leaving leaves and a muddy residue as well as soaked walls, now-kaput appliances and a massive number of drenched items.

My wife and I both are accumulators. Indeed, our daughter-in-law long has argued we should start getting rid of stuff, I guess fearing she’d be stuck doing that job in due course. The basement was full of shelving units containing all sorts of things, including toys our son played with decades ago (he certainly had lots of LEGO sets!), personal memorabilia, collectibles such as some of my typewriter collection, parts for my vintage Studebaker, silk flowers my wife uses to create seasonal arrangements, vintage electronics like Betamax video recorders, books, and humdrum household supplies.

Unfortunately, the flooding reached items on the first shelf of the shelving units and floated large storage containers off the floor, buffeting their contents.

So, we’ve spent an enormous amount of time and energy since the storm going through everything that

had been in the basement, throwing away loads of priceless, irreplaceable items, decontaminating anything salvageable as well as trying to repair what we could. (For instance, I devoted last night to painstakingly prying open some fancy kaleidoscopes, to clean residue from prisms and lenses.)

Meanwhile, a crew now is removing ruined portions of wallboard, etc., and then will start reconstruction.

Fortunately, New York City has stepped up its services. The city sent workers, dressed in hazmat suits, to remove items we wanted out of the basement that we couldn’t manage ourselves. They “schlepped” two refrigerators to the street and also brought up some large heavy storage boxes full of toys and other contaminated possessions. The Sanitation Department has added extra pickups and is taking everything put out, no matter how much. The city even has arranged for a variety of food trucks to provide free lunches to people in the neighborhood.

Now, don’t get me wrong. I’m not looking for sympathy. I certainly realize people in areas like Louisiana and even New Jersey have suffered far worse, losing their homes and virtually all their belongings.

Instead, I want to stress the lesson I’ve learned — the need to consider rethinking where and how to store things you consider irreplaceable. Once our basement is repaired, the bottom shelves of the shelving units only will contain items we don’t worry too much about losing. ●



Bottom shelves only will contain items we don’t worry too much about losing.

MARK ROSENZWEIG, Editor in Chief
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Why Doesn't Your Mill Work Well?

Figuring out how to get the most from your equipment can be a grind

PARTICULATE SOLIDS are atoms or molecules that either are arranged in ordered arrays (crystalline), have no ordered structure beyond the short length scale (amorphous or glass), or are polymeric. Naturally, each material will exhibit its own characteristic physical properties. Disregarding the class of solids, the grinding process involves breaking bonds between individual atoms or molecules that comprise a solid. The breakage results from internal tensile or shear stresses established by the application of some external force. Grinding equipment apply various modes of external stress:

- impact,
- compression,
- shear, and
- abrasion.

Impact breaks particles based on the force applied by other particles or the impact surface. To some extent, the resulting particle size distribution (PSD) follows the existing cracks in the starting material. The force applied is limited because new particles are released following Rittinger's Law (energy required is proportional to increase in surface area).

Compression relies on the elastic modulus to reach the yield stress and generally gives a narrower PSD because most of the force goes to the larger particles.

Shear tends to break chunks of the starting particles off from the parent material. When the internal stresses in the starting material are high (low fracture toughness), these chunks can crumble into finer particles. Shear usually is a poor use of energy because the surface-to-volume is lower than the previous modes of applied stress.

That's where abrasion comes in. The three comminution mechanisms already mentioned have a lower limit of size whereas attrition can produce the finest particles. Attrition or erosion is a process in which a wear surface removes the atoms or molecules. Unfortunately, the wear surface often takes the worse beating. When the particle hardness is more than 20% greater than the surface hardness, we invariably get high wear. Comminution equipment usually favors one mechanism while using all of them in the overall size reduction.

The extent of fracture generally depends upon the applied method of external stress and the magnitude of this stress for a given material. It's important to appreciate that the material's physical

properties also strongly impact the extent of fracture. In grinding equipment, the dominant method of stress application is governed by the equipment design. Consequently, understanding the fracture mechanics of a given material is crucial for specifying the correct type of equipment or determining why a mill doesn't work properly. Sometimes we want to avoid these modes of external stress — specifically to preserve particle size (i.e., eliminate dustiness).

Materials fall into three classes: brittle, ductile or semi-brittle, with the last the most common. The physical properties of primary interest include elastic modulus, Poisson's ratio, yield stress, hardness and fracture toughness. These are related to some extent. As an example, the ratio of yield stress and hardness is a fixed value for a given material. Usually hardness is three times the yield stress. Materials that break easily are brittle and have a high hardness and low fracture toughness. The most difficult material to grind is a ductile material because the yield stress is low and crack propagation does not occur easily. We had a polymeric chemical that was so ductile it smeared in every device that was tried. The plant had some success with a ball mill — but it took a long time to get the desired size. A short study found the hardness became much higher at lower temperatures. A pin mill worked very well when liquid nitrogen was injected during milling. Basically, we had converted this ductile material to a semi-brittle one.

While theoretical models for comminution processes exist, I generally would refrain from using these for design. I'd rather rely on the physical properties as a guide in the choice of a piece of equipment. Past experience, taking into account the physical properties of previous materials and the equipment used, is the key to selection. Given their sparse resources, most plants face difficulties in obtaining such details. So, the best bet usually is to discuss the application with established equipment manufacturers; talk to more than one because their experiences will differ and their recommendations will reflect the products they offer. (If all you have are hammers, everything looks like a nail!) ●

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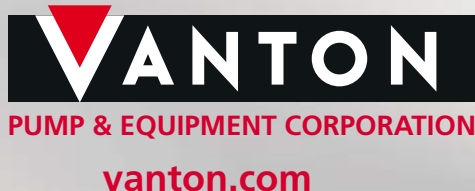
Rely on the physical properties as a guide for equipment choice.

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Properly Vet Contractors

Some sensible steps can stave off unwelcome surprises

ENGINEERS ONCE flew around the country to check out contractors' facilities before signing deals. Nowadays, many bosses instead heavily rely on the Internet. Unfortunately, a nifty-looking web site can make a firm seem impressive and much more capable than it actually is. You don't want to discover the truth after a contract is signed.

So, let's consider how to vet contractors, engineers, and consultants. First, understand their particular scope of work (SOW). The SOW should define skills very specifically. Second, make sure their bid is detailed; it must identify the skills required. Third, because the first level of defense in managing project cost is the contract manager, ask one question: "Does the bid make sense?"

Do a careful cost analysis yourself and revisit it once the bids come in. Each firm must give enough details in its bid to permit an honest evaluation. Many will be reluctant to provide labor rates. Use data from RSMMeans to define labor hours and require the companies to provide labor costs based on a carefully defined cost schedule of the work to be completed. In practice, this can offer valuable insights. For instance, a plant I worked for needed a contractor to demolish a salt pit and construct steel to support equipment above. I knew from the analysis that three bidders understood the job; the fourth clearly didn't because its costs were way too low for the demolition.

CHECK OUT PAST FIELD NOTES

More than a decade's worth of real-world tips are available online at www.ChemicalProcessing.com/field-notes/

For additional practical pointers, check out the online roster of Plant InSites columns at www.ChemicalProcessing.com/plant-insites/

Now, let's look at some traps to avoid in the vetting process. One of these is past performance; wealthy contractors and engineering firms can afford to scrub the Internet of their mistakes. Also, don't regard a backlog of work necessarily as a sign of a good or competent firm, contractor or fabricator. Here's another one: don't put too much weight on another engineer's recommendation of a firm — for several reasons: 1) past performance is a quantitative thing based on a particular manager; 2) each job is unique; 3) the

capabilities cited may no longer exist or reside only in a specific satellite office; and 4) the engineer making the recommendation may view a project from a different perspective than you do, say, based on mechanical equipment, while you focus on instruments, leaving you open to blind spots in managing the work.

In fairness to contractors, many SOWs fail on their own lack of merits. You never should expect a contractor to do an assignment that is fuzzy on details, requires more hours than you can ask your management to assign, or doesn't break down tasks by discipline. A common ploy is to slap a "not-to-exceed" label and live with an incomplete SOW — but this only leads to over-runs. In the end, your company may unfairly blame the contractor for its laziness or incompetence.

Fuzziness often reflects lack of experience. I once explained to a boss that he didn't know what was involved in running 800 ft of threaded pipe because, unlike me, he never had done it. Remember your limitations and don't make off-the-cuff assumptions.

As for the "requires more hours than you can ask your management to assign," I am referring to detailed work such as updating piping and instrumentation diagrams. Assigning exact hours for these types of efforts is very difficult. They best are completed in-house.

Now that we've discussed why past performance is a poor measurement of a contractor's qualifications, let me present some vetting tools you may want to consider. Bonding and insurance often are brought up but I'm an engineer, not an insurance expert; so, you might ask for your insurer to lend a hand. Investigating a firm's safety record is tough unless it has gotten citations; instead look at its shop — tools that are put away in gangboxes tell a story, so does general cleanliness and wearing of safety glasses. Perhaps the best option is to ask questions: 1) Does the firm have contingencies? 2) Does it have other jobs to assign to workers during delays? 3) Has it worked with sub-contractors before? 4) What equipment must it rent? And, most important, 5) does it have any questions? Lastly, investigate the company's financial health, if possible; watch for red flags, e.g., spurts in cash flow; a low cashflow/debt ratio; and declining income.

In the end, vetting only goes so far. Our business is still about a handshake. ●

DIRK WILLARD, Contributing Editor
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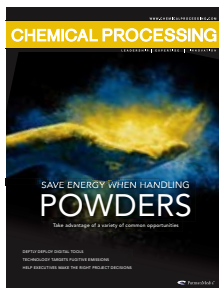
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I just had to write and say what a kick I get out of your publication, especially this last one [December 2019 issue, [http://bit.ly/2q\]wDJL](http://bit.ly/2q]wDJL)]. [Editor's note: we only run a Letters column infrequently, hence the delay in when this and the next comments appear.]



I teach the capstone Chemical Process Design class for chemical engineers at UC [University of California], Berkeley each Fall. I just finished my fourth year of this, but I just hit 40 years in industry last summer (Dow Chemical, J&J, Novartis, and several pharmaceutical start-

ups). I figure my role is to not just teach the basics of design to these very bright but very naive students, but also to give them a taste of industrial issues and problem-solving — things that should help them no matter where they end up.

This issue hit on several things from authors that I want to emphasize:

Dirk, I liked what you said about watching people work and looking for problems. [See: Field Notes, “Don’t Stop at the Drawings,” <http://bit.ly/30WTNdr>.] Don’t just sit in your office and look at what the computer tells you (see below).

Alan, your hydraulic survey story is just like one of mine where a sleepy-looking operator solved a steam flow bottleneck that baffled all of our engineers. [See: Energy Saver, “Don’t Get Tangled Up by Complexity,” <http://bit.ly/2RXSk2c>.]

Andrew, that was a great example of used equipment not being a bargain. [See: Plant InSites, “Is Buying Used Equipment Really a Bargain?,” <http://bit.ly/30Z6i89>.] I teach my students my “Baker’s Dozen of Rookie Design Mistakes” and that’s one of them!

Maybe all of you would appreciate the response I got on the final exam, where I posed a safety incident and had them propose instruments that could have prevented it, then asked if they had any other thoughts. About one third of them wrote to “go look in the damn tank.” Maybe when I get better at this I’ll get to where all of them say that!

George Tyson
University of California, Berkeley

REMOTE ENGINEERING EDUCATION MAKES SENSE

Interesting article. [See the September 2020 issue, From the Editor, “Can ChE Students Avoid School Daze?,” <https://bit.ly/3wcWdme>.] Based on my recent work experience, I believe the best answer is video conferencing, which can accommodate both lectures and one-on-one interactions.

Chats and screen sharing are also great tools usually available to most video conferencing tools. I have been working from home for months now, using these tools, and our team productivity is on a par with our previous in-the-office experience. It is challenging learning new tools, but that in itself is a great introduction to the current work environment: evolving new software tools and new procedures is an everyday experience in the ChEng practice.



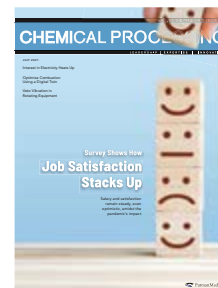
Alfredo Saettone
Engineer Principal - Process
Global Engineering

THE CSB NEEDS EXPERTS

I agree with the statement that it would be best if at least one CSB board member had practical experience with process safety from the chemical or related industries. [See: CP’s July 2021 edition, “Add Industry Perspective to the CSB,” <https://bit.ly/3xdz3ND>.]

However, when I think back on the Baker panel, I don’t think Secretary Baker had any practical process safety experience but, in my view, the result of his investigation in the Texas City refinery explosion was excellent.

I have read many CSB reports — but not all — and in my view the conclusions or recommendations often don’t focus on the persons responsible for the company, i.e., directors and boards. Too many, in my view, focus on regulations and guidelines. So maybe a lack of practical experience could be good for the outcomes of the investigation, so other leaders in the chemical industry see the need for actions to improve process safety.



Niels Jensen
Retired chemical engineer

More Efficient Heat Exchangers Emerge

Topology software and additive manufacturing lead to markedly better performance

SIGNIFICANT IMPROVEMENTS in heat exchangers now are possible because recent advancements in 3D printing allow designs previously thought impossible, note researchers at the University of Illinois at Urbana-Champaign, Urbana, Ill. They have coupled metal 3D printing with software tools using topology optimization to create more-efficient, compact tube-in-tube heat exchangers.

“We developed shape optimization software to design a high-performance heat exchanger,” explains William King, professor of mechanical science and engineering at the university’s Grainger College of Engineering. “The software allows us to identify 3D designs that are significantly different and better than conventional designs.”

Pairing the shape optimization software with additive manufacturing, the researchers designed fins internal to the tubes in a tube-in-tube heat exchanger. The team then 3D printed the tubes using a powder-based metal additive manufacturing process.

“Any material that can be made with powder-based metal additive could be used. There are dozens of these materials available today and the list continues to grow,” says King.

“The additively manufactured tubes can handle the same service range as conventionally manufactured tubes. The

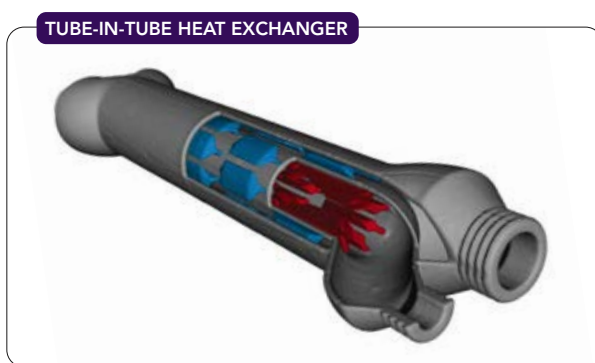


Figure 1. Computer tomography X-ray image displays hot fluid (red) in the outer tube and cold fluid (blue) in the inner tube. Source: Hyunkyung Moon, Davis McGregor, Nenad Miljkovic and William P. King.

pressure and temperature range could go much higher because of the materials available in metal additive manufacturing,” he points out.

“Our optimized heat exchanger has about 20 times higher volumetric power density than a current state-of-the-art commercial tube-in-tube device,” adds Nenad Miljkovic, associate professor of mechanical science and engineering and co-study leader.

Details appear in an article in the journal *Joule*.

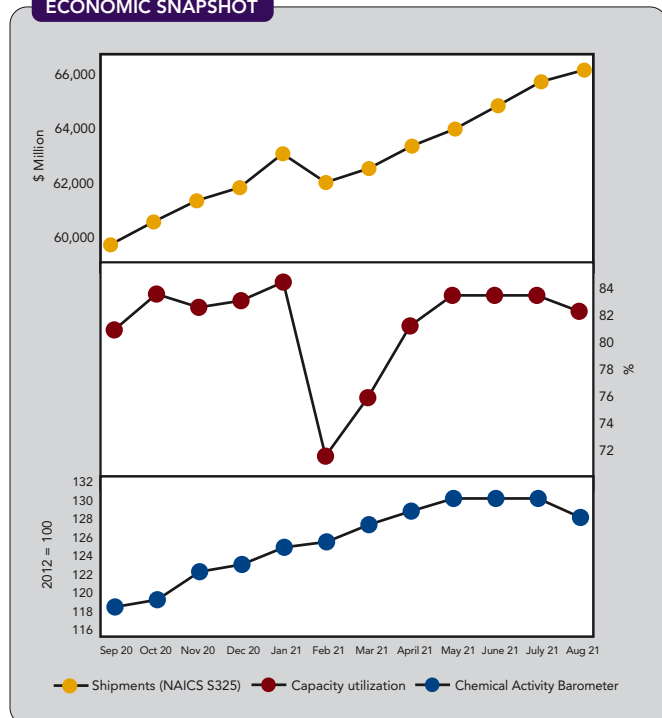
The optimized device also performs well compared with shell-and-tube and brazed plate heat exchangers designed for significantly higher heat transfer. “There is a great potential to innovate on the sizes and shapes of heat exchangers, using additive manufacturing to create new three-dimensional shapes that can lead to high performance. Over time, we will see these types of innovations impact all types of heat exchangers and applications,” believes King.

The compactness of the tube-in-tube exchanger offers a variety of savings, e.g., in fluid inventory and support structure requirements, among other advantages. “In designing this heat exchanger, we were considering mobility applications — electric cars and electric airplanes — as real opportunities for compact heat exchangers. There are other benefits, for example small heat exchangers use less fluid and less pumping power. They also are smaller to store and transport, and use less material leading to additional sustainability benefits,” elaborates King.

The tubes created by 3D printing also are extremely robust, note the researchers. “The materials are quite strong. Modern additive manufacturing can produce metal parts with high strength, close to zero porosity, and low surface roughness,” King stresses.

However, like with any heat exchanger, small passages can lead to fouling, he cautions. “The fouling issues would

ECONOMIC SNAPSHOT



Shipments rose slightly but capacity utilization and the CAB slipped. Source: American Chemistry Council.

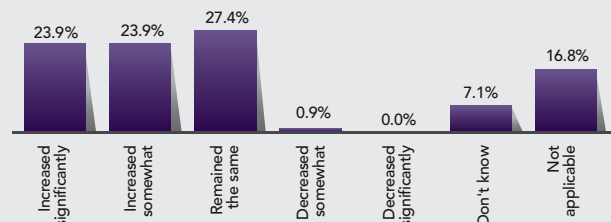
need to be managed the same way that one would do for a conventional heat exchanger with the same sized passages.”

The approach extends to other types of heat exchangers as well: “Using generative design to find high-performance, three-dimensional shapes, and additive manufacturing to realize these shapes could be applied to any type of heat exchanger including shell-and-tube heat exchangers and plate-and-fin heat exchangers,” says King.

“Just about every company making heat exchangers is closely monitoring the development of additively manufactured heat exchangers, because there are significant performance benefits that can be unlocked. There are only a few commercial applications of additively manufactured heat exchangers, but these will increase over time,” he concludes. ●

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How has the level of female engineers
at your site changed in the past three years?



More respondents reported an increase versus no change or a decrease in the number of female engineers on their sites.

Electrocatalytic Route Eases Urea Production

MANUFACTURE OF urea relies on the well-established two-stage Haber-Bosch process. However, a highly selective single-stage electrocatalytic route may challenge that method, hope scientists in Singapore. Their patented process, which reacts nitrate ions and carbon dioxide at ambient conditions with an indium hydroxide catalyst, provides comparable efficiency to the Haber-Bosch method while avoiding its severe operating conditions.

The process boasts yield and catalytic selectivity that are up to 40% higher than other electrocatalytic routes to urea production, says team leader Alex Yan, a professor at Nanyang Technological University's School of Materials Science and Engineering, Singapore.

“Our method essentially manipulates the chemical reaction process to become highly selective,” he adds.

Faradaic efficiency, nitrogen selectivity and carbon selectivity reach 53.4%, 82.9% and almost 100%, respectively. This efficiency matches that of the Haber-Bosch process, according to Yan and his team, but without its need for pressures approaching 200 atmospheres and temperatures up to 450°C.

The researchers also found the catalyst suppresses hydrogen evolution while creating a low energy barrier for the all-important C-N coupling (Figure 2).

An article in a recent issue of *Nature Sustainability* describes their work.

Yan and his team currently are striving to refine catalytic selectivity to speed up reaction time. They also plan to develop a prototype to demonstrate scaled-up urea production.

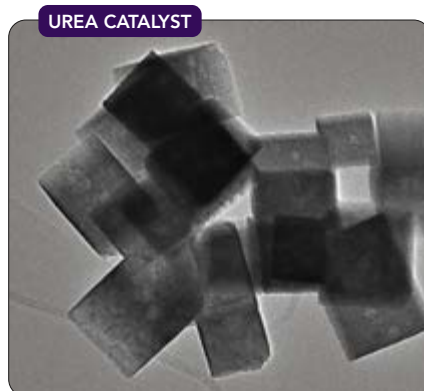


Figure 2. Indium hydroxide electrocatalyst boosts Faradaic efficiency, nitrogen selectivity and carbon selectivity. Source: Nanyang Technological University.

“This prototype is still at the stage of laboratory research. To realize scaled-up urea production through the sustainable electrocatalysis route, three main issues should be addressed: urea yield, catalyst stability and selectivity,” he notes.

These three factors also would pose challenges to commercial-scale production, although Yan believes the main chemical and engineering challenges could lie in the eventual extraction and purification of urea from the electrolyte.

A solar-powered prototype also interests the researchers. “The idea is

intriguing but we have not started relevant work yet. However, urea selectivity is dependent on applied potential, so the photoelectrochemical approach might be feasible,” Yan explains.

The simplicity of the process eventually could lead to its adoption at both large and small scales, he feels, perhaps with agricultural areas generating their own urea for fertilizers from mobile or modular units.

While the new process suggests an appealing route for urea production, it also provides a deep insight into the underlying chemistry of the C-N coupling reaction that could guide the sustainable synthesis of other indispensable chemicals, he stresses.

“Many important chemicals might be produced through electrocatalytic C-N coupling reaction, such as methylamine. We have not investigated any other chemicals yet, but we may do so if we clearly elucidate the C-N coupling mechanism in urea synthesis,” Yan concludes. ●

Design in a Pinch

Overly complicated pinch processes can reduce achievable savings



The use of pinch analysis must be tempered with realism.

PINCH ANALYSIS is a powerful tool for designing heat recovery systems. I provided a brief introduction in *Chemical Processing's* July 2019 issue, "Take the Heat Off Pinch Analysis," (<https://bit.ly/3ES2A3I>). That column focused on pinch targets. Today, I'd like to move on to pinch design.

First, a quick recap: Process streams that are cooled down, releasing heat, are called "hot streams." Those that are heated, absorbing heat, are called "cold streams." Note that "hot" and "cold" do not refer to the temperature of the streams. Rather, they denote the direction of heat transfer.

When we examine heating and cooling demands in most processes, there is a "pinch," characterized by a "pinch temperature" that divides the process into two distinct regions:

- The region above the pinch temperature has a net heat deficit. An external utility heat source (e.g., steam or a furnace) completes the energy balance.
- The region below the pinch temperature has a net heat surplus. An external utility heat sink (e.g., air or cooling water) removes the excess heat.

To minimize the utility heating and cooling requirements, and thus achieve the pinch "energy target," we must design independent heat integration systems for these two regions. This is the basis of the pinch principle: *Do not transfer heat across the pinch.*

If you don't transfer heat across the pinch, you are guaranteed to achieve the energy target for the process. However, designing heat recovery systems isn't always easy, even for seemingly simple systems.

Pinch analysis was initially developed to improve new plants. Following the pinch principle, the design approach starts by separating the streams into two groups: those above the pinch temperature, and those below it. A systematic procedure is then followed, whereby heat transfer matches are created between the hot and cold streams in the above pinch group, until all available heat is consumed. At this point, at least one cold stream will be short of heat; the deficit must be satisfied by a utility heat source.

Following the same approach for the group of streams below the pinch will result in at least one hot stream with excess heat that will need removal by a utility heat sink. However, this

systematic procedure often results in complex heat integration designs, with large numbers of heat exchangers and multiple stream splits (i.e., process streams that are divided into two or more parallel branches). These designs generally require simplification to ensure operability or to reduce costs. Consequently, most practical designs use somewhat more energy than the pinch target.

Pinch analysis also applies to revamps. In these cases, the design technique needs significant modification for several reasons. In most revamps, strong incentives exist to maximize use of any existing heat exchangers, even if they are not ideal when viewed from a pinch perspective. This often results in revamp designs that markedly differ from new plant designs. Revamps must account for existing equipment and plot space. This can limit the opportunity to add new heat exchangers. Finally, revamps typically occur during turnarounds, when time is at a premium. This makes it hard to justify complex projects that are difficult or time-consuming to execute.

Many different retrofit design procedures have been proposed. Most approaches start by identifying the existing heat exchangers in which heat crosses the pinch. The various approaches then use different methods to correct these inefficiencies. However, as in new plant designs, these methods often lead to overly complex designs, thus reducing achievable savings. The resulting revamps typically include modifications to existing heat exchangers (e.g., tube bundle replacements, to increase heat transfer capability), installation of new heat exchangers as well as piping to change the way existing heat exchangers are connected.

Pinch analysis is a powerful tool, though its use must be tempered with realism. Properly applied, it can often reduce process heating and cooling requirements by 15% or more. ●

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Is EPR on the Rise for Packaging?

Law adopted by two states makes producers responsible for product waste

ON JULY 13, 2021, Maine became the first state to enact Extended Producer Responsibility (EPR) legislation for packaging. On August 6, 2021, Oregon followed, enacting a similar EPR law applicable to packaging. Other states are poised to pass similar legislation. This article discusses the concept of EPR and summarizes the state legislation.

WHAT IS EPR?

EPR has been around a long time. It is the concept that the original producer of a material bears responsibility for it beyond post-consumer use. In the European Union, it is a much more mature concept and business norm. In the United States, EPR is viewed chiefly as a funding mechanism to support recycling programs by shifting the responsibility to pay for these programs from municipal and other public sources to private product producers. EPR embeds a version of “polluter pays.” Many U.S. organizations have actively espoused EPR for more than 15 years, but industry pushback has slowed adoption of mandated EPR programs like those recently enacted in Maine and Oregon.

U.S. EPR programs for certain product categories, including paint, carpet, batteries and pharmaceuticals, already exist. However, mandated programs have not targeted other product categories, like plastics and packaging, until now. Several factors have converged to jump-start these packaging EPR initiatives. Unsurprisingly, the costs to maintain recycling programs in general have increased dramatically. Second, the pandemic has generated vastly more single-use packaging waste; the imperative to recycling such waste has drawn more public attention to the issue, which predated the pandemic but which COVID-19 greatly exacerbated. Finally, the complexity of the packaging waste stream has increased substantially; some materials are not as amenable to recycling as others. This has incentivized lawmakers to craft mandated solutions to these problems; shifting the cost burden to the producer is widely seen by some as an efficient fix.

NEW EPR PACKAGING LAWS

The Maine law focuses on plastics and packaging materials. The law requires product producers to fund stewardship organizations that collect, recycle and otherwise manage end-of-life products manufactured by the producers. The legislation extends to most packaging materials, but includes exemptions for

paint and beverage containers, small businesses, and low-volume packaging producers, all terms defined in the law. While the law is intended to incentivize the use of materials that are easily recycled, its primary purpose is to transfer the cost of managing and recycling packaging waste to the product producer.

The Oregon law creates an EPR program that includes several “covered products,” including packaging, printing and writing paper, and food serviceware. Covered products specifically exclude several categories of products, including bound books, beverage containers, napkins, paper towels, and specialty packaging used exclusively for industrial or manufacturing processes, among other categories. Under the law, producers of these materials must join stewardship organizations, called producer responsibility organizations, or PROs, that must assess annual membership fees based on the environmental impacts of the producers’ products.

TAKEAWAY MESSAGES

EPR may well be a concept whose time has come in the United States. With state and local resources severely stressed by pandemic demands, eroding tax bases, and competing priorities, state legislatures with a more progressive leaning can be expected to welcome opportunities to shift the cost burden of recycling programs to others. Some believe that burden is appropriately directed to the producers of the material generating the waste.

Whether this is the start of a national packaging EPR trend or something else is unclear. It seems clear, however, that product manufacturers would be wise to recognize that consumers increasingly expect businesses to be accountable for the environmental consequences of their products and their recycle or reuse in demonstrably more defined ways. In other words, even in the absence of legislative directives, consumer expectation likely will influence business practices. State initiatives similar to the Maine and Oregon laws probably will proliferate; the types of materials covered by such initiatives could well expand. Businesses will need to monitor these initiatives carefully and understand the supply chain, cost, and business reputation implications of such potential legislation. So, too, will financial entities investing in these businesses and the products they market. ●

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Consumers expect businesses to be accountable for the environmental consequences of their products.



YVAN MASSON
Executive Vice
President of BIAR
sampling systems

If you use a system that has absolutely no dead space, then no purging and flushing is needed.

Safe Sample Systems Bolster Bottom Line

Grab representative samples in a simple way the first time, every time

GRABBING SAMPLES of hazardous chemicals in liquid and gas states is challenging but necessary. Manufacturers rely on quality samples to ensure products specs and operators rely on this equipment to help them grab those samples. But not all sampling platforms are the same. Chemical Processing spoke with Yvan Masson, Executive Vice President of BIAR sampling systems, a manufacturer of liquid and gas sampling systems, to learn how the right sampling system can make a big difference for a plant's bottom line.

Q: How can sampling valves improve chemical plant safety and efficiency?

A: Certain processes are just not sampled because it's too difficult to do so. But if you're able to measure and take corrective action in the middle of the process, you're more effective and efficient because you're not waiting until the end to realize that your production is not within the specs. I've heard operators having to grab a sample with a scoop or a bucket directly from the top of the reactor. To do so, they have to wear heavy PPE and the whole process itself is outdated; it's not safe and it's not efficient.

Q: That sounds dangerous. How can safety be improved?

A: You have to understand what sampling is. First of all, you have to get a quality sample. A quality sample means a representative sample. And to get a representative sample, there are only two ways to do that. The traditional way is by purging and flushing the equipment and then grabbing a representative sample. It's a bit like turning on the tap, letting some product flow to the drain, and then collecting your sample in a clean bottle. I know that's very rudimentary, but even the most sophisticated closed-loop systems that allow the process to be done without exposure require purging, flushing and dealing with the waste.

Another and better way is to get a directly representative sample the first time every time. If you use a system that has absolutely no dead space, then no purging and flushing is needed. Minimizing hazardous product handling increases safety, reduces waste and environmental emission. It also improves efficiency.

Q: Why just one sampling valve at a time? Why not go all in?

A: Well, that's a very good question. From our point of view, it'd be great if customers would decide to change all devices at one time. While sampling is important, more often than not, this is not the priority. There are other priorities like pumps and control valves. Also, if it's not broken, why fix it? And that sometimes is the mentality. What I suggest to customers is it's better to start where the biggest impact can be seen — the most challenging sample point. Our range of sample valves is not necessarily for every plant or every unit within those plants, but for those units that put priority on safety, sample quality, industrial hygiene and the environment. Also, to be effective, operators have to trust the new system. We highly recommend picking the most challenging sample point and doing something about it. Once the operators see how easy it is to use an inline sample valve as opposed to a sample panel, for example, they'll beg maintenance and management to replace all sample points. Most plants are still utilizing technology that has existed since the 1950s. While these systems may have advantages, in



Figure 1. Simple, robust solution helps eliminate exposure and improves industrial hygiene and environmental compliance.

many cases they were not designed with safety or modern standards of industrial hygiene in mind. Challenging the status quo and adopting a new product, or just a different product as a matter of fact, is often considered risky. Our product has been in use since the late 1970s throughout Europe and the rest of the world and has a proven track record when it comes to reliability.

Q: What are the differences between traditional sample panels and what you're offering at BIAR?

A: Sample panels have multiple fittings and valves and are complicated to use. It requires opening the tap and purging and then it goes to the flare. The new sample panels are sophisticated and they allow the whole process to be done without exposure. But for that, you need nitrogen to push the liquid, you need a vent, you need a flare connection and so on. Typically, there are a multitude of valves to operate, and this has to be done in a precise sequence to get the job done in a safe and effective way.

Imagine if the plant alarm goes off while the operator is halfway through sampling; what does he have to do? Well, he or she has to go to the nearest shelter-in-place. And what happens with the sample panel if it's halfway open, halfway purged? It's very complicated. That's one of the problems. The main difference with our systems is they are robust and simple to use. They are very intuitive and there's only one valve to operate. It's an inline valve, and as standard, our valves come with a spring-to-close handwheel, also known as a dead-man's handle, which springs back and shuts the valve should anything happen. So on the one hand you have a system that is complicated, not very robust and inefficient and on the other, you have a simpler, safer and more efficient system. So it's a big difference.

Q: Are the BIAR sampling systems ideal for every sample need?

A: BIAR sample valves are most suitable for applications where the following are important: sample quality, industrial hygiene and safety. As a rule of thumb, the more hazardous the application is, the better fit it will be. If the product is not hazardous or is very easy to recycle, then a simple tap or ball valve may be a better option from a capital cost point of view.

Many facilities don't know that they have a problem. They've been sampling for years with outdated technology and they think their way is the only way. But at the end of the day if the operator is wearing a lot of PPE and has to go back out and grab a second sample, it adds exposure risk and doubles the time necessary to take a sample. We ask our customers, How do you sample? Do you have to purge and flush? And if you do, would it make a difference if you didn't have to purge and flush? If the answer is yes, then

PODCAST ACCESS:

Listen to the entire conversation via podcast at <https://bit.ly/GrabSample>.



we can work with you to find a solution to see if it's cost-effective to replace the existing installation with one of our inline sample valves.

Q: How do you justify the investment?

A: There are different types of sample valves out there and some are more cost-effective than our systems. While they might be cheaper from a capital cost point of view, the running cost is something that is often forgotten. It's only after a couple of years, you realize you've already paid two or three times the cost of capital in terms of maintenance and spare parts. So that's one of the reasons to go with a robust inline valve that requires very little, if any, maintenance at all.

Q: Are there any disadvantages with a BIAR sample valve?

A: From a capital cost point of view, our sample valves may be higher. And that really is the biggest disadvantage. Also, if the product is easy to recycle and is not very hazardous and doesn't contaminate the environment, then maybe you'd be better off with a ball valve and purging and flushing. But, even if it is easy to recycle waste, is it not better not to have any in the first place? Maybe it's worth looking into a solution like ours. Not to mention the safety aspect. PPE should not be the first barrier of protection, it should really be the last. And that's what we're trying to achieve with our sample valves. They might be a little bit more expensive than a ball valve or a piston-type valve, but they offer extra protection and less exposure for the operator.

Q: Is there anything you'd like to add?

A: There are only two ways to grab a representative sample, and representative samples are the most important point of sampling. If you grab only 100 milliliters or a couple of ounces of sample out of a batch that has thousands of gallons, then those couple of ounces have to represent what you have in your reactors. The only two ways to get a representative sample are by either purging and flushing or by getting a directly representative sample the first time every time. So ask yourself, which one is better? Which one is more efficient? It's better to have a product you can trust; lab technicians will be happy because they have reliable samples and plant management will be grateful because their plant is running efficiently and effectively.

For more information visit: www.biar.us

FOUR PRODUCTS WIN VAALER AWARDS



Diverse innovative offerings earn laurels

By Mark Rosenzweig, Editor in Chief

Chemical Processing has bestowed Vaaler Awards biennially for more than 50 years. These awards recognize products that promise to significantly improve the operations and economics of plants. This year's awards were open to entries that were commercialized in the United States between May 2019 and June 2021.

The awards are named after John C. Vaaler (1899–1963), chairman of *Chemical Processing's* Editorial Board from 1961 until his death. He became the publication's editor in chief in 1946, after 24 years in the chemical and related industries.

An impartial panel judged the entries. It consisted of the members of *Chemical Processing's* Editorial Board — a group of seasoned technical

professionals with diverse responsibilities and from a variety of industry sectors (see sidebar). The panel scored all entries on three criteria: technical significance; novelty or uniqueness; and breadth of applicability. The judges weren't required to give any awards — but did choose to honor four products:

- Centurion guided-wave-radar level transmitter from Hawk Measurement, Medina, Ohio;
- Fluid Genius digital platform from Eastman, Kingsport, Tenn.;
- Interceptor-QV passive isolation device from CV Technology, Jupiter, Fla.;
- Style 2848 compression packing from Teadit, Pasadena, Texas.

Level Transmitter Features PoE Communications

Power-over-Ethernet (PoE) communication offers a number of advantages, including secure and reliable in-plant and off-site monitoring as well the capability for remote sensor set-up, diagnostics and troubleshooting. The Centurion Guided Radar (CGR) level transmitter (Figure 1) from Hawk Measurement is the first and only guided-wave-radar (GWR) unit with PoE communication. It enables monitoring of tank levels from smart phones, tablets and computers.

Commercialized in the United States in January 2021, the CGR level transmitter can measure the level and interface of liquids, sludges, powders and granules up to 124 ft. Its GWR technology provides a response time of <1 sec and an accuracy of ± 3 mm; pressure or vacuum, temperature, viscosity, foam, dust, coating of the probe, or changes in dielectric constant don't affect it. The device can send tank alarms and alerts via email and directly to smart phones.

If troubleshooting is required, service technicians need not enter a facility. Instead, the CGR PoE instrument can send information about its health for off-site diagnostics and, if necessary, reconfiguration.

The device can connect to an online portal, the Hawk-Eye365, to enable monitoring of multiple tanks worldwide in real time. The online portal provides access to critical data, including volume, material height, historical trending, alarms and alerts, sensor set-up and diagnostics. Control-room and operations-level screens can be tailored to operating company specifications; they share a common graphical user interface and can have permissions set for different tiers of employees.

The CGR level transmitter features dual interface capabilities and built-in digital displays. It can auto-calibrate to any dielectric ≥ 1.5 . The device is rated for hazardous gas and dust services by IECEx, FM, CSA and ATEX.



Figure 1. Guided-wave-radar device features power-over-Ethernet communications.



Platform Optimizes Heat-Transfer-Fluid Performance

Many process operations must operate at a specific temperature. Plants generally rely on heat transfer fluids to provide and maintain the desired temperature, which can range from -100°C to $+400^{\circ}\text{C}$. Such fluids usually are robust but can deteriorate due to thermal stresses and service conditions unique to the particular process. Fluid Genius (Figure 2) from Eastman is a patent-pending digital platform that provides a data-driven analytical approach to help engineers preserve optimal fluid quality and forecast predic-

tive maintenance. Plant staff can access the secure web-based platform from any device with a modern browser.

Launched in the United States in May 2021, Fluid Genius uses artificial intelligence, fluid-specific degradation kinetics, and key parameter thresholds to predict a fluid's life expectancy and advise on how to extend life while avoiding unplanned shutdowns. It works for almost any organic heat-transfer-fluid system.

Fluid Genius provides a fluid condition score, which is a unique indicator of overall fluid condition, and sends alerts about possible fluid contamination.

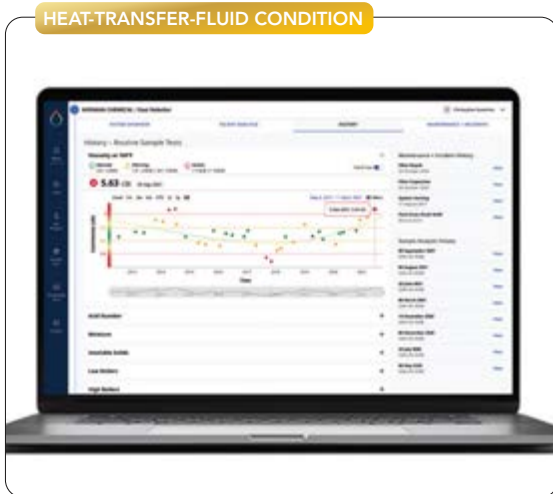


Figure 2. Web-based platform provides insights on fluid state and desirable predictive maintenance.

The easy-to-use technology also will generate notifications and fluid trends as well as give customized recommendations for critical actions, such as system venting, inert-gas-blanketing system installation and inspection, fluid replacement, and implementation of side-stream filtration. In addition, it affords easy access to order and manage sample analysis kits.

The application, which contains nearly 50 years of operating-system sample-analysis data, draws in-depth insights on fluid chemistry behavior over time. Those data, coupled with end-user input into its maintenance and incidence log, and advanced artificial intelligence/machine learning techniques, enable the platform to provide proprietary analytics for fluid replacement and budgeting.

THE JUDGES

- Vic Edwards, process safety consultant, Houston
- Fred Gregory, process safety and risk manager, Lubrizol, Deer Park, Texas
- Rachelle Howard, associate director of automation and controls, Vertex Pharmaceuticals, Boston
- Darren Moroziuk, manager of manufacturing support, Pfizer, Kalamazoo, Mich.
- Julie O'Brien, director of sustainability, Air Products and Chemicals, Allentown, Pa.
- Roy Sanders, process safety consultant, Lake Charles, La.
- Ellen Turner, market development representative, Eastman Chemical, Kingsport, Tenn.
- Dave Vickery, director of product management, Aspen Technology, Midland, Mich.

Passive Isolator Removes Air-Recycling Risks

Recycling clean air from dust collectors and pneumatic conveying systems can minimize the need for makeup air and, with that, ease maintaining the desired air temperature and humidity level. However, the risk of a dust explosion propagating through return lines into an occupied area prompts many plants to vent clean air returns to the exterior of the building, necessitating installation of extensive duct runs. Isolating the lines can prevent such propagation. The Interceptor-QV from CV Technology is an isolation device based on the principles of flameless venting that can allow air recycling while providing continuous protection. It has no moving parts or need for regular maintenance.

Introduced in the United States in February 2021, the Interceptor-QV (Figure 3) is a passive device that operates automatically and doesn't rely on an electromechanical element. Instead, a stainless-steel-mesh cartridge removes energy from the flame front of the deflagration as it passes through the tortuous path of the mesh, quenching the deflagration and preventing any flame from passing beyond the device. Cartridge replacement does not require removing the device from the process.

The Interceptor-QV uses a patented system in which a differential pressure switch continuously monitors pressure drop across the mesh cartridge, alerting operators to any buildup of dust. Another patented feature is an integrated thermocouple that, via a relay, indicates its exposure to the intense heat of a deflagration.

The quench valve comes in four sizes and can handle clean-air return-line applications with pipe-line or duct diameters from 2 in. to 100 in. It is ATEX certified as an explosion isolation device.

PASSIVE ISOLATION



Figure 3. Device uses mesh to provide continuous, automatic and maintenance-free protection of return air lines.

Traceable Packing Meets Stringent Fugitive Emissions Standards

Regulations and operational imperatives are spurring plants to continually strive to reduce fugitive emissions from control and isolating valves, pump shafts and other equipment elements. Style 2848 compression packing (Figure 4) from Teadit is a certified low-emission packing that is self-lubricating as well as resistant to heat, pressure and chemical attack. It features "Teadit tags," particles inseparable from the packing that enable easy identification as well as full traceability even after use. In addition, its metal-free composition resists high installation stresses. The packing can successfully handle a wide range of process environments (-240°C to +455°C, pressure to

COMPRESSION PACKING

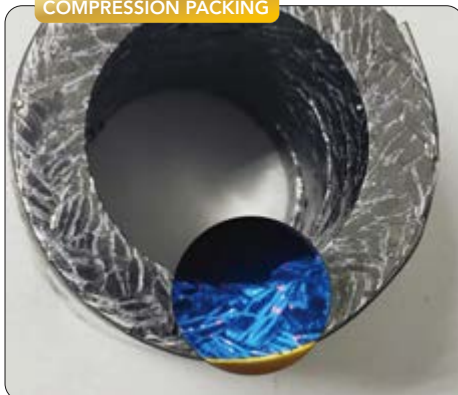


Figure 4. High-performance braided packing incorporates tags that create a uniquely identifiable fingerprint.



255 bar, and 0–14 pH) and media while still meeting the most-stringent fugitive emissions requirements.

Style 2848, which debuted in the United States in January 2021, incorporates a proprietary yarn made from flexible graphite reinforced with a polytetrafluoroethylene structure embedded with the Teadit tags. These tags allow identifying the packing even when both packing and valve tags are missing; this permanent identification makes the packing ideal for applications requiring a warranted product. The packing's design lowers friction, which improves control valve accuracy and may enable use of smaller actuators, and meets strict industry standards (ISO 15848-1, EN 16752 and API 622 - 3rd ed.), while also avoiding the issues posed by the metallic reinforcement commonly used in this type of product.

For control valves, the packing maintained less than 2 ppm leakage even after 100,000 mechanical cycles and four thermal cycles — per ISO 15848-1's most-stringent test

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procedure, CC3. Tests on isolating valves done according to API 622, 3rd ed., showed leakage below 2 ppm even after 1,510 mechanical cycles and five thermal cycles. ●

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Motor Efficiency Turns Upward

Regulation and end-user desires are spurring improvements

By Seán Ottewell, Editor at Large



THE ECO Design regulation of the European Union (EU), which came into force on July 1st, mandates that all drives marketed in member countries must fall within the high efficiency classification (IE2) set by the International Electrotechnical Commission (IEC), Geneva, Switzerland, in IEC 60034-30-1—and be marked IEC compliant; the IE2 efficiency requirements are 25% lower energy losses than a reference value. The new regulation also calls for a wide range of motor packages to meet the even higher requirements of the IE3 (premium efficiency) class. In July 2023, the regulation expands even further, raising the base level in certain cases to IE4 (super premium efficiency) class. These efficiency requirements relate to the entire electric-motor-driven system, not just the motor itself.

The regulation covers a large segment of low-voltage drives (LVDs), 0.12–1,000-Kw output power, single AC voltage output drives, for example standard variable speed drives (VSDs), and voltages from 50 V to 1,000 V.

ECO Design is just the latest step in a process of transition to even greater efficiency that is expected to save the EU 102 TWh of electricity annually by 2030.

FIVE MAIN IMPACTS

Vendors such as Rockwell Automation, ABB and Siemens are finding their chemical industry customers largely geared up for these changes. However, many important issues are at play. Andy Gagnon, senior manager engineering, mechanical and control hardware, Rockwell Automation, Mequon, Wisc., stresses the latest legislation:

- affects all manufacturers that label drives for global use and adopt IEC standards;
- applies in any country that has adopted IEC (or similar) ECO Design requirements;
- helps customers know a drive meets current best-in-class efficiency design, materials and construction;
- offers ease in applying LVDs with confidence of getting the best efficiency; and

- includes a standard method for publishing efficiency information for application considerations.

“I really like the ‘helps customers’ point because we finally have a methodology to calculate a drive’s efficiency relative to a standard reference value,” he notes.

“For the first time with drives, we have standards and are making sure everyone adopts them. Another beautiful part of the standard is that it prescribes for us as drive manufacturers how we should calculate energy losses. So you can make direct comparisons between drives supplied by different vendors,” he adds.

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However, process engineers need to understand some of the subtleties within the new standard, Gagnon cautions. For example, it’s very important, he notes, to account for any losses incurred by an AC or DC link choke incorporated as part of a drive’s design and the impact on the efficiency the drive ultimately can deliver as it operates that motor. Engineers also should be aware of exclusions to the regulation, including regenerative drives, DC drives and bus supplies and drives integrated into products such that testing their energy efficiency separately is not possible.

Another advantage of the standard is that it overcomes the issue of multiple points of efficiency for drives.

“One of the challenges that has beleaguered the industry for a long time was that drives innately can control motors at different speeds and different load points and oftentimes that has challenges in terms of understanding what that means in terms of efficiency. The IEC standard language is RCDM — the reference complete drive model

— which helps customers find the best way to apply any drive to meet the standard,” says Gagnon.

BETTER DECISION-MAKING

For its part, Rockwell Automation provides an online ECO Tool for finding energy efficiency details of its Allen-Bradley line of LVDs. Today, the tool supports PowerFlex 750 series drives (Figure 1); coverage of additional lines of PowerFlex drives will follow in the future.

“A customized tool like this really lets you understand the energy efficiency of motors and drives. For chemical companies, it clearly shows the relationship between power use, need and cost. So you could look at them as a good return on investment [ROI],” emphasizes Gagnon.

Steffen Zandler, heavy industry strategy and marketing manager, EMEA, Karlsruhe, Germany, agrees: “Chemical and other process companies increasingly are looking to get help from a sustainability and optimization point of view anyway — wanting to know what and where they can tweak to make their processes more efficient.”

Even with this detail at hand, additional factors still can come into play, notes Gagnon: “Harmonic distortion, for example, can lead to heating issues and poses challenges for motors and upstream devices. So power quality matters.”

Active and passive filters can tackle the problem. However, an active front end with regeneration technology offers potential energy savings by “taking energy normally burned off as heat in the resistor and making it part of the utility feed,” he explains.

Benefits of regeneration include: obviating braking resistors; eliminating cooling equipment and associated wiring, labor, installation and maintenance costs; and the possibility of returning energy for use in other applications or to the utility.

“That’s where this ECO directive is designed. So we get better at thinking about having the best-in-class hardware in our drives and best-in-class processes around drive control and we can optimize losses in the drive as it runs across its operating range,” declares Gagnon.

“Regeneration in particular has significant added effects and should be considered when customers are looking to create a powerful energy consumption roadmap that meets sustainability requirements and, ultimately, the bottom line. At the end of the day, this stuff costs money. You don’t get energy for free,” he concludes.

NO SURPRISE

“Generally speaking, our experience has been that customers in the chemical processing industries were well aware of the coming changes well in advance of them coming into force. So, we didn’t really have to pursue them,” says Jukka Hannuksela, head of global standards and compliance for ABB IEC LV motors, Vaasa, Finland.

“In fact, in Germany, the VIK standard was already requiring IE3 before the EU regulation was published. However, when talking about the market and customers they are not always the same across Europe. It is probably fair to say that end customers have a slightly greater awareness of the need to adopt higher IE classes than OEMs [original equipment manufacturers],” he adds.

Indeed, ABB finds some end users are striving to exceed the efficiencies mandated by the regulation and, so, are searching for even-more-energy-efficient technologies for their equipment, applications and processes.

“That is a good indication of the common ‘industrial’ interest to reduce energy consumption,” stresses Hannuksela.

Of course, the quest for greater energy efficiency extends well beyond the EU. For instance, the United Kingdom will adopt the same regulation, although by a different name, Hannuksela notes. In the United States, almost all motors are covered by the integral horsepower motor final rule, which calls for premium efficiency levels equivalent to IE3. China uses an energy efficiency grading system, with the highest — 3 — comparable to IE3. Australia, New Zealand and some South Pacific countries also have adopted the same or similar requirements.

The IE4 classification doesn’t represent the top practical efficiency. Indeed, ABB’s latest offerings are designed to surpass the 2023 regulations. The company says its IE5 “ultra-premium efficiency” motor packages represent the highest level of efficiency achieved by any current design. In practice, IE5 motor packages have 20% less energy losses compared to IE4 ones.

A good example of such an IE5 motor package is ABB’s SynRM. Opting for such a unit not only puts users a step ahead of the regulations and reduces energy consumption but also provides considerable savings. An IE5 motor package, comprising a motor and a VSD, will pay back the cost difference with an IE3 package after about 13 months, according to the company.



Figure 1. Online tool now provides information on the energy efficiency of this and other drives in the same series. Source: Rockwell Automation.

Future EU mandates might take a broader focus. There have been discussions to require “system level” energy-efficiency requirements and regulation, Hannuksela notes.

“So the future trend could be for the combination of electric motors and VSDs and, perhaps, also including a whole system covering, for example, pumps. The question of how the energy efficiency of the full combination would be evaluated and assessed in industry applications may require some further thinking and standardization,” he says.

“Overall, I would stress that moving to more-efficient motors, ideally in combination with VSDs, improves the performance of machinery and reduces operating costs. At the same time, of course, it benefits the environment — which is the purpose of the regulation,” he adds.

The regulations pose under-appreciated dangers to OEMs, warns Hannuksela. “Unless they are fully aware of the implications, OEMs specifying low-voltage motors and VSDs in their products risk missing, misunderstanding or misinterpreting important details. This could potentially lead to them producing unsaleable equipment.”

BROADER DISCUSSIONS

Thomas Niedermeier, lead, low voltage motors for Siemens Digital Industries, Ruhstorf an der Rott, Germany, also has noticed growing interest by EU-based chemical companies in finding ways to improve the efficiency level of motors used in their processes.

“In the EU chemical industry, more than 75% of all motors are located in hazardous area zones, so the companies always insist on having types available that meet the required standards. Generally speaking, it’s often this industry demand that has been propelling suppliers such as Siemens to move towards compliant motors even before it became prescribed by law,” he says.

In general, large markets like China already are following IEC rules or, sometimes, holding themselves to even higher standards, Niedermeier notes. So, in Siemens’ case, the company’s motors fully comply with different international demands.

As an example of the value of using more-efficient motors in combination with VSDs to improve machinery performance and operating costs, Niedermeier cites a current Chinese project.

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Figure 2. High-voltage motors control and optimize compressor-train performance on new polypropylene lines at Chinese site. Source: Siemens.

Oriental Energy, Zhangjiagang, China, now is commissioning two 600,000-t/y propylene plants. Both use a propane dehydrogenase process.

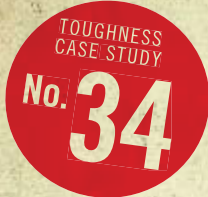
“To run our plant economically, flow through our reactors has to be precisely controlled. Having an efficient and powerful compressor train with a highly

reliable motor drive system is crucial for our process and for our business,” notes Zhou Y Zhong, general manager, Oriental Energy.

At the heart of the process are three compressor chains controlled by a highly efficient Siemens motor system. The compressor trains are built from Simotics high-voltage motors in combination with Sinamics GL150 low-frequency converters that control and optimize the motors in the compressor chain (Figure 2). This tandem design, which enables feeding power to the motors from two sides, is the first of its kind in the world, according to Siemens.

“At the end of the day, our productivity is increasing because everything is running reliably,” adds Lou Wen Yao, Oriental Energy chief electrical engineer.

Over time, discussion in industry will evolve from focusing solely on motors to a broader perspective that considers complete systems, Niedermeier believes. “From an environmental perspective, there is limited advantage to be gained from improving efficiency just for motors. We need to look at the bigger picture and optimizing efficiency in integrated systems,” he concludes. ●



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CONSIDER PRINTED CIRCUIT HEAT EXCHANGERS

These units' small channels offer high efficiency in clean services

By Amin Almasi, mechanical consultant

Source: Heatric (a division of Meggitt UK Ltd.)

PRINTED CIRCUIT heat exchangers (PCHE) are a relatively recent option for process heat transfer. They get their name from the chemical milling procedure used in their manufacture, which is the same process employed for printed circuit boards. Introduced into the market beginning in the late 1980s, these compact exchangers feature flat metal sheets with flow channels chemically etched into them. The plates then are stacked and diffusion bonded to form a solid metal block containing precisely engineered flow passages. The channels typically are 2-mm semicircular in cross section. The small channel size fosters exchanger compactness.

Fluids can move in parallel-, counter-, cross-flow, or a combination of these to suit the requirements of the particular service. The operating temperature and pressure drop constraints for the given duty govern the required configuration of the channels on the plates for each fluid set. The channels can be of unlimited variety and complexity.

PCHEs can operate at a wide range of temperatures from cryogenic (say, -196°C) to above 900°C. They can handle a broad variety of clean fluids and find use in many different processing applications requiring high efficiency and performance.

ADVANTAGES

PCHEs boast thermal efficiency on the order of 98%. Their high heat-transfer surface area per unit volume results in reduced weight, space and supporting structure. As a rough

indication, PCHEs are four to six times smaller and lighter than conventional exchangers such as shell-and-tube units. In some cases, the reduction in size and weight compared to a shell-and-tube exchanger has exceeded 70%. For instance, a PCHE weighing 11 metric tons replaced a shell-and-tube exchanger weighing more than 70 metric tons.

The lighter and more compact exchanger greatly decreases costs for foundations, support structures, piping works, installation, operation and maintenance. In addition, the PCHEs' far lower fluid inventory than conventional exchangers can offer many safety and reliability advantages. For instance, it enables substantially smaller sizes of pressure relief devices and relief discharge piping compared to those of an equivalent shell-and-tube exchanger with far larger inventory of fluids.

The channels of PCHEs are optimized for the flow pattern and thermal duty. This results in a very efficient and high-performance heat transfer. Another advantage is the reduction in energy waste. PCHEs can be used for gases, liquids and two-phase flows. Approach temperatures as close as 2°C typically are achievable within a single counter-flow exchanger, thereby avoiding the need for multiple exchangers or shells.

The fabrication technique — diffusion welding, which also is called diffusion bonding — used for PCHEs gives a high integrity homogenous heat exchanger. Diffusion welding involves the migration of atoms across the joint due to concentration gradients. The two materials are pressed

together at an elevated temperature usually between 50% and 75% of the melting point. The pressure is used to relieve the void that may occur due to the different surface topographies. In other words, this special welding process promotes grain growth between the surfaces. Under carefully controlled conditions, diffusion-bonded joints reach parent metal strength. Stacks of plates are converted into solid blocks containing the fluid flow passages. No gaskets or sealing are needed inside a PCHE. The risk of leaks is approximately two orders of magnitude lower than for any other conventional heat exchanger. This is a great operational, reliability and maintenance advantage.

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PCHEs can be made from many materials, including different grades of stainless steels, alloy steels, titanium alloys and austenitic nickel-chromium-based superalloys, etc.; some of these alloys are oxidation- and corrosion-resistant materials well suited for service in extreme environments subjected to high pressure and extreme temperature. The compactness of a PCHE allows effective use of expensive materials and special alloys. (As with any exchanger, material selection must consider thermal, mechanical and physical properties, corrosion and environmental resistance, fabricability, availability and cost.)

DISADVANTAGES AND LIMITATIONS

PCHEs sometimes are more expensive than other options such as shell-and-tube units, mainly due to their need for all-stainless-steel construction at a minimum — unlike shell-and-tube exchangers, which often can extensively use carbon steel. In addition, while the exchangers can handle corrosive and exotic fluids, these fluids must be extremely clean and free of any debris, solids, etc.

A well-known limitation is the pressure drop developed in a PCHE for low-pressure-drop, large-volumetric-flowrate applications. Pressure drop is roughly inversely proportional to the channel diameter. For high-pressure applications, pressure drop might not be a constraint but it can be a barrier for low- or moderate-pressure applications. Each flow channel acts like a small pipe with many bends; swirl flows, reversed flows and eddies occur around a bend corner. The reduction

of hydraulic diameter, complexity of routes and other effects can lead to a channel pressure drop that's unacceptably large.

PCHEs should be engineered and fabricated to be immune to flow-induced vibration and other dynamic effects. In fact, properly designed PCHEs had no failure due to these issues. However, some low-cost PCHEs that were badly engineered and perhaps poorly manufactured have experienced flow-induced vibration in certain situations — despite PCHEs having been proven to be very resistant to pressure fluctuation by their inventor. So, the design and fabrication of PCHEs should take this into account.

Blockages can occur easily due to the fine channels. The high risk of blockage posed in many applications requires installation of fine filters or strainers at the inlet of their PCHEs. Units have been operated for many years in numerous so-called clean fluid services without issues. Including a working filter or strainer as part of the exchanger set-up is mandatory for commissioning and strongly recommended for normal operation in non-clean duties. Of course, these filters demand regular cleaning, which adds to operating and maintenance costs.

It is far better to order the filter or strainer as a part of the PCHE package from its manufacturer rather than to get it independently. (Some manufacturers offer a range of high-integrity inline conical strainers.) Standard off-the-shelf filters or strainers might not suffice for such a special application. The recommended maximum strainer aperture is approximately one third of the minor dimension of the flow passages. As a very rough guide, a PCHE needs a fine filter or strainer of 200, 250 or 320 microns. The filter or strainer should be installed as close to the exchanger inlet nozzle as possible. After commissioning, the strainer should be cleaned, checked for integrity and then reinstalled for the normal operation. The pressure drop should be measured across the strainer element independently of the heat exchanger. This allows monitoring of any particulate build-up and scheduling of cleaning before any damage occurs.

Any blockage that occurs will call for various cleaning methods ranging from high-pressure jetting to advanced and expensive chemical cleaning. This can be difficult in some installations — so, cleaning nozzles and access must be considered to facilitate these operations. Every PCHE system should provide for the possibility of such cleaning methods as part of good operational practices. Galvanic compatibility with the piping material and others has caused some difficulties; an insulation kit or coated spool piece may be needed for on-site installation.

ENGINEERING AND OPERATION

PCHEs can handle extreme temperature and difficult services needing high integrity and effectiveness across different operating conditions. Optimizing a unit involves

finding the best geometric variables and operating parameters for the particular application. This requires consideration of both thermal and hydraulic performance.

Many modern PCHEs feature zigzag flow channels. These zigzag flow paths do not allow boundary layer growth and encourage turbulent flow. By enhancing heat transfer area and increasing local flow velocity at channel bending points, the zigzag channel shape improves heat transfer performance compared to exchangers with straight or other simple pattern channels. Wavy geometries such as zigzag ones provide little advantage at low Reynolds numbers; maximum advantage is at transitional Reynolds numbers. For higher Reynolds numbers, the free shear layer becomes unstable; vortices roll up, thus enhancing heat transfer. At high Reynolds numbers, periodic shedding of transverse vortices and other effects raises the Nusselt number with a considerable increase in the friction factor. Some experts have suggested double-faced configuration. However, this is very difficult to implement due to plate alignment, especially when considering large plates on a tall stack — particularly if the flow paths are complex.

S-shaped patterns offer another option. The Nusselt number of PCHEs with zigzag patterns is 25–35% higher than those with S-shaped patterns — but the pressure-drop is about 2.5–5 times larger, depending on Reynolds number. Properly optimized S-shaped models theoretically decrease pressure drop to 25–35% of that of conventional zigzag models while reducing heat transfer performance only slightly. They particularly would suit applications with pressure-drop constraints. However, S-shaped PCHEs need much more expensive etching and challenges exist for proper diffusion bonds.

Headers usually are half cylinders that enable fluid distribution between the nozzles and the channels in the exchanger core. This allows formation

of fluid distribution areas inside the exchanger core. Headers and nozzles are welded to the core to direct the fluids to the appropriate sets of passages. There are a variety of methods for supporting PCHEs. Most commonly, end-type

supports welded to the exchanger core are used. Alternatives include saddle supports fitted to the lower header in the vertical plane.

Commissioning of PCHEs demands great care. More attention than usual

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must be paid to the cleanliness of associated piping and equipment. Thorough cleaning is essential — as is flushing and draining of the whole system to ensure removal of any scale, corrosion products, debris, etc.

COMPARISON TO PLATE HEAT EXCHANGERS

Plate heat exchangers (PHEs) are popular compact alternatives to shell-and-tube units. They come in many different types and constructions. The most common type consists of a series of channel plates pressed together to form a plate pack. Gaskets used in such PHEs impose some limitations and lead to high maintenance costs and even some operational problems. Gaskets, which must be compatible with operating fluids, often restrict the operating temperature. On the other hand, the gasketed construction allows dismantling for maintenance or even adding more plates if needed. PHEs suit a wide range of clean and quasi-clean services (for instance, cooling water applications).

The diffusion-welded construction of PCHEs, while avoiding the limitations of gasketed PHEs, is far more

expensive — and enables them to handle extreme operating temperatures. However, their solid structure, while eliminating any maintenance issues posed by gaskets, prevents dismantling like conventional gasketed PHEs. So, PCHEs only make sense for extremely clean services that pose absolutely no risk of dirt, debris, scale, etc.

APROPRIATE APPLICATIONS

Because PCHEs typically are more expensive, they cannot replace conventional heat exchangers in all services (even not all clean services). Two typical areas of application are:

1. Where space and weight matter. Light and compact PCHEs can offer great advantages, e.g., in many revamp, expansion and renovation projects.
2. Where process requirements dictate the application of PCHEs. This can include services with small temperature differences between flows or demanding higher efficiency and performance in heat transfer. ●

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Select the Proper pH Sensor

Consider a number
of key factors including
the value of digitalization

By Nikodem Siwek, ABB

ACCURATELY MEASURING the levels of acidity and alkalinity in chemicals production is vital to ensuring the highest standards of product quality, safety and environmental performance. Thus, pH is one of the most widely checked process parameters. However, it also is one of the most difficult to get right. The choice of a suitable pH measurement device depends upon the nuances of the particular process and the nature of the substance involved.

Chemical processors face many issues that require managing pH. For example, reactions and other production steps often demand careful pH control. In addition, the need for pH measurement usually extends to effluent streams; the wrong pH value can lead to serious corrosion in the pipework carrying the waste, especially at older facilities built with less-corrosion-resistant materials. Furthermore, lack

of correct balancing of pH levels at the point of discharge could cause damage to the aquatic environment, potentially resulting in prosecution and stiff penalties.

For process engineers, the wide range of sensor options available (producers of pH electrodes often have extensive and complex portfolios) can make selection difficult. Exacerbating the situation, the retirement of

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EASIER MONITORING



Figure 1. Digital technologies provide new opportunities for operators to check the pH performance of their processes.

more-experienced engineers has left many operating companies with less expertise on pH measurement.

The latest developments in the digitalization of pH measurement help address all these issues. Today, pH devices are easier to install, commission, operate and maintain. The latest ones offer “plug and play” technology that allows fast connection of the sensor to a digital transmitter, cutting the time needed for installation and removing any uncertainty during commissioning.

Digital technologies allow operators to check the pH performance of their processes from a tablet (Figure 1) or another smart device. The inclusion of features that help simplify operation and maintenance also enables engineers to quickly get to the root of any functional issues. For example, many instruments can produce a dynamic QR code to indicate faults. A technician can use a smart phone to scan this code to get diagnostics information and the possibility of online help from the vendor.

Digitalization also can provide a better overview of processes, ensuring more informed and objective decision-making. Integrating the readings of pH sensors and others into digital management systems makes data collection more efficient, saving both time and costs, while affording a more practical way to control overall plant operations.

GETTING STARTED

A key preliminary for selecting the right pH electrode for an application is understanding the process itself. Because processes often involve fluids that range from mild to highly caustic or acidic as well as conditions that can vary in temperature, a device should offer the best balance of durability in the particular operating environment and

performance. Therefore, knowing exactly what is being measured can help set sensible criteria for device selection.

In addition, it aids to have a grasp of the factors that can affect pH measurement performance. This requires a basic understanding of how a pH electrode functions.

Today’s devices work on an electrochemical principle. A sensor known as a glass pH electrode is used in conjunction with a reference electrode to complete an electrical circuit that produces a pH value for a measured sample.

A basic glass electrode comprises an inert glass stem sealed to a bulb or membrane made from a special glass formulation that is responsive to hydrogen ions.

The pH measurement results from an ion exchange process that takes place between the hydrogen ions in the solution and the ions at the surface of the glass membrane. This develops a charge on the membrane surface that then is transferred through the membrane where it is picked up on the inner surface.

The glass electrode (Figure 2) contains an aqueous internal filling solution of a known pH along with a silver wire coated with silver chloride (AgCl), which is called an internal element. The immersed element allows for electrical continuity with the inner surface, thus affording an electrical connection back to the pH meter.

To complete the electrical circuit, a reference electrode provides a return path to the sample solution. Reference electrodes come in various designs but a typical construction uses an AgCl-coated silver wire immersed in a potassium chloride (KCl) solution. This offers a stable environment for the reading but, equally important, allows for an electrical continuation between the pH electrode and the sample, thus completing the circuit.

More-demanding applications, such as those involving sulfides, require use of a double reference electrode. Such a reference electrode usually consists of a AgCl sealed electrode with its own junction, fitted into a second chamber with a junction in contact with the sample. The main advantage of this electrode is that the reference solution in the second chamber, usually just KCl, can be chosen to be compatible with both the “inner electrode” solution and the sample. This electrode can have a slurry-filled sealed outer chamber or a reservoir-fed arrangement to suit the application.

Once the fundamentals of a pH electrode are understood, it becomes easier to grasp what can go wrong. In general, pH electrodes have several potential weak points that

can limit both their effectiveness and overall service life if not considered at the outset. So, let's look at three key aspects:

1. *The electrode glass.* The formulation of the glass used for the electrode can significantly impact its performance, both in terms of accuracy and ability to withstand extended exposure to the inherent process conditions. Some processes involve substances that can be very aggressive, subjecting the glass pH electrode to prolonged attack that can accelerate wear. For example, glass and semiconductor processing often relies on solutions containing hydrofluoric acid (HF). HF etches the surface of glass membranes, eventually dissolving them away completely. In this situation, opting for an HF-resistant glass will lengthen the working life of the electrode.

Caustic processes with pH levels of pH 12 or above also can pose problems. Here, sodium ions can exceed the concentration of hydrogen ions, causing a sodium error that results a reading lower than the true pH value. The resolution is to choose a glass type that offers a low sodium error.

GLASS ELECTRODE



Figure 2. The pH sensor contains an aqueous internal filling solution of a known pH along with a coated silver wire called an internal element.

In addition, the temperature of the sample being measured can affect the performance of the glass. In situations where either the medium itself is at a low temperature or the sensor is installed in low-temperature conditions, opting for a low-temperature glass will help ensure a fast response to changes in pH. Conversely, in high-temperature processes where media are more aggressive, using a high-temperature glass will aid in protecting against premature ageing of the glass that can quickly degrade the performance of electrodes with general-purpose pH glass.

Equally as important is the design of the glass electrode. For example, a glass with a self-cleaning flat profile will help reduce the risk of fouling in applications with high levels of particulate matter. Alternatively, bullet glass sensors are the prime choice for any application up to 140°C and 10 barg. Their robust construction suits in-line, dip and retractortype installations in a variety of applications.

2. *The reference electrode.* To ensure accurate performance, it is essential that the reference electrode potential is very stable and not affected by chemical changes in the solution. Most pH sensors use an Ag/AgCl reference electrode containing a chloridized silver wire immersed in an electrolyte solution of KCl. This solution slowly seeps out of the sensor through a reference junction to provide an electrical connection between the reference element and the sample.

The solution also includes AgCl to help stop the coating on the reference element from dissolving.

A common problem with reference electrodes is poisoning caused by the ingress of chemicals such as sulfides and bromides from the sample being measured. Over time, poisoning can change the chemistry of the reference electrode, creating reference potential instability and reducing the accuracy of the pH measurement. It can decrease the lifetime of the electrode, necessitating early removal and replacement with a new one.

3. *The reference junction.* This provides the interface point between the reference electrode solution and the process sample. To ensure effective measurement, the solution must flow freely through the junction to mix with the sample and establish the electrical circuit.

The design of the reference junction can play a major role in helping reduce the risk of electrode poisoning and providing prolonged stability and resistance against fouling. Making the path between the sample and the reference as long and complicated as possible can significantly extend the operational life of a sensor. Some electrode types offer designs such as multiple junctions — for example, ABB's 500PRO electrode boasts a triple junction design — and options such as solid-state reference technology, where a material such as wood charged with KCl serves as the reference, can aid in avoiding problems with plugging and poisoning that can affect liquid-, gel- or slurry-filled electrodes. Other options include designs that feature a longer path between the reference junction and



the electrode as a way of delaying the impact of any poisoning from the sample.

Under certain circumstances, such as where the reference system becomes contaminated by salts evaporating out of the electrolyte or where the sample itself contains substances that can form salts, the reference junction can become either blocked or fouled, restricting the flow of the solution and impeding the measurement.

Various options are available to help minimize the risk of blocking. For instance, junctions made from polytetrafluoroethylene (PTFE) offer good protection against the formation of particulate matter; PTFE junctions are ideal for most applications except those involving hydrocarbons. For those types of applications, a better alternative is a solid reference junction using a substance such as wood impregnated with KCl. Less prone to becoming blocked by hydrocarbons, a solid reference can help prolong an electrode's lifetime and improve its long-term performance.

INSTALLATION CONSIDERATIONS

Many manufacturers offer different installation options for their pH instruments. So, knowing the measurement location can be useful. Ensuring that a pH electrode is placed in the right part of the process can make a material difference to its performance. In particular, to prevent the sensor from drying out, it should be in constant contact with the sample medium.

Given the variables that can affect a pH device's performance, it's sensible to locate the sensor to enable easy access for inspection and carrying out maintenance tasks such as cleaning and calibration.

How a sensor is installed also can massively impact its performance and operation. For example, mounting the sensor to a tank or other vessel can prompt problems because flow within the vessel can be omnidirectional and cause accelerated fouling. Locating the sensor in a recirculation line can deliver the benefits of a "self-cleaning" mechanism due to the unidirectional flow of the sample, which will help keep the sensor operational for longer.

Depending on the application and the medium being measured, a variety of installation options are available to help facilitate access; these include retractable systems for high pressures, flow cells and dip-type sensors.

THE IMPORTANCE OF DIAGNOSTICS

Many pH electrodes now offer extensive diagnostic data on the status of the device. This information can help identify both deteriorating performance and its possible root causes.

The advent of digital pH systems, in particular, has enhanced these diagnostic capabilities. Because an applica-

tion typically requires a large number of pH sensors, being able to quickly identify the particular device that is failing can help save both time and engineering resources. Modern digital pH instruments have built-in software, making them very quick and easy to install, configure and maintain. It now increasingly is possible to dial into devices to find out everything you need without having to physically visit them. Many advanced digitally enabled measurement devices can check internal connections and electronics as well as warn of sensor memory failure.

Digitalization has markedly enhanced the process of diagnosing a fault. For instance, issues such as electrode poisoning now are easier to trace thanks to features like perpetual impedance diagnostics (found on ABB's next generation pH and ORP sensors), which analyzes the resistance and impedance between the reference and measuring electrode; and smart reference electrode monitoring, which provides early warning of electrode poisoning, enabling quick diagnosis of problems.

Condition monitoring can lead to the optimum scheduling of maintenance and allow advance preparation to avoid process impact. By connecting the pH sensor to a digital transmitter, the unit's data output from its diagnostic functions can help accurately determine the root cause of a pH measurement error. A plant can use these data to identify what went wrong and derive strategies to prevent the same problems from recurring. Operations and maintenance staff can make an informed decision on whether the fault is fixable or the sensor needs replacing — minimizing downtime and reducing the need for unnecessary inventory.

MAKE THE RIGHT CHOICE

With so much to consider when it comes to pH electrode selection and the extensive array of different options offered by manufacturers, determining the optimum choice can seem daunting. Hopefully the pointers in this article will help. Fortunately, some manufacturers are trying to ease decision-making.

For example, ABB recently launched a simplified portfolio with just five distinct ranges of dedicated electrode options designed for specific duties, from general purpose through to harsh and ultra-pure applications. Each range incorporates the necessary features to address many of the challenges outlined earlier, enabling users to find the right device for their requirements more easily and quickly. ●

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High-Throughput Experimentation Qualifies Commercial Catalyst

Parallel testing eases selection of gas-phase-hydrogenation catalyst to load during turnaround

By Enrico Lorenz, Tobias Zimmermann, Alexander Higelin, Moritz Dahlinger and Joachim Haertlé, hte; Nilenindran S. Govender and Sachin Teli, Sipchem; and Abdulaziz A. Almathami, Prince Mohammed Bin Fahd University

MAXIMIZING PROCESS efficiency to ensure competitiveness and improved margins continuously drives large-scale production of industrial chemicals. The catalyst is a key component of many of these processes. It not only requires tuning to achieve maximum activity but also tailoring of its physical and chemical properties to the surrounding process. High product selectivity avoids wasting valuable feedstock and protects the downstream section from energy-intensive separation and pollution of the equipment with side products. Furthermore, the catalyst must withstand feed impurities and provide flexibility with regard to process conditions.

Upgrading a process during a turnaround with the latest-generation of the catalyst or even switching to an alternative technology offers significant opportunities to increase plant profitability but also involves technical and economic risks. This article describes how high-throughput catalyst performance evaluation can support chemical production plants in quantifying the potential gains and mitigating the risks early on in the catalyst selection process.

Sipchem, Al Khobar, Saudi Arabia, a globally recognized manufacturer of base chemicals, intermediates and polymers, cooperated with hte, an independent catalyst tester based in Heidelberg, Germany, to evaluate commercial hydrogenation catalysts for the gas-phase conversion of dimethyl maleate (DMM) to butanediol (BDO). Performance results of the laboratory-scale test were in line with the commercial-scale reference plant, and stability data supported Sipchem's decision for catalyst replacement.

PARALLEL TESTING PROGRAM

Continuous gas-phase hydrogenations of high-boiling substrates often operate under a great excess of hydrogen, with molar H_2 :substrate ratios typically exceeding 50:1. Examples include DMM to BDO, fatty acid methyl esters to fatty alcohols, aldehydes to oxo alcohols, and nitro aromatics to aromatic amines. Such hydrogenations require enormous hydrogen flows and represent a challenge at both commercial and laboratory scale. Commercial catalyst design must provide low pressure drop and high

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mechanical strength to deal with the high flow rates. Furthermore, the design of the internal pore structure must prevent capillary condensation [1,2].

The commercial catalyst system tested here was a stacked bed with a guard layer (top) and a conversion layer (bottom). hte developed a customized packing protocol to ensure defined and reproducible reactor filling; hte can handle shaped materials, fibers or powders within high-throughput setups [3].

The catalyst was crushed and sieved to a certain particle fraction and physically mixed with an inert to achieve

a plug-flow pattern and avoid channeling. This allowed control of the pressure drop and fostered isothermal operation by uniform heat distribution along the catalyst bed in stainless steel reactors with an inner diameter of 5 mm and a length of 400 mm.

hte supported Sipchem’s experimental program and the objectives of the study to test the impact of process conditions while still considering individual catalyst

effects. The sophisticated reactor filling concept enables conducting several experiments in parallel. Here, these involved 1) residence times variation (achieved by loading different catalyst masses); 2) testing of spatial catalyst arrangements; 3) changes in the mass ratio of guard to conversion bed; and 4) duplicate positions to verify the rig performance and data quality. hte uses a fully integrated digital laboratory workflow based on

proprietary software. Its myhte relational database combines all available catalyst and reaction data to allow advanced structure performance evaluations.

hte used a 16-fold high-throughput gas-phase system for this project. It serves reactors with inner diameters of 4 mm to 15 mm to test materials from powder scale up to commercial shapes. Mass flow controllers and syringe pumps provide precise dosage of gas feeds and liquid components, respectively. Each of the 16 reactors received the same amount of hydrogen and methyl ester feed. A block heater controlled the temperature and furnished a uniform 15-cm-long isothermal zone (with a maximum temperature deviation of

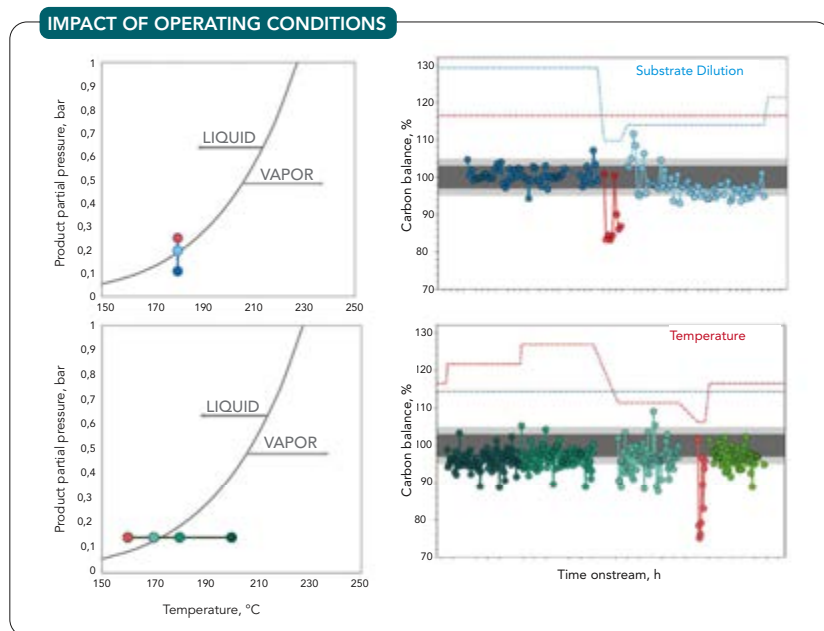


Figure 1. Graphs show carbon balance for different feed dilution ratios and reactor temperatures in comparison to the phase diagram of butanediol as the high-boiling product.

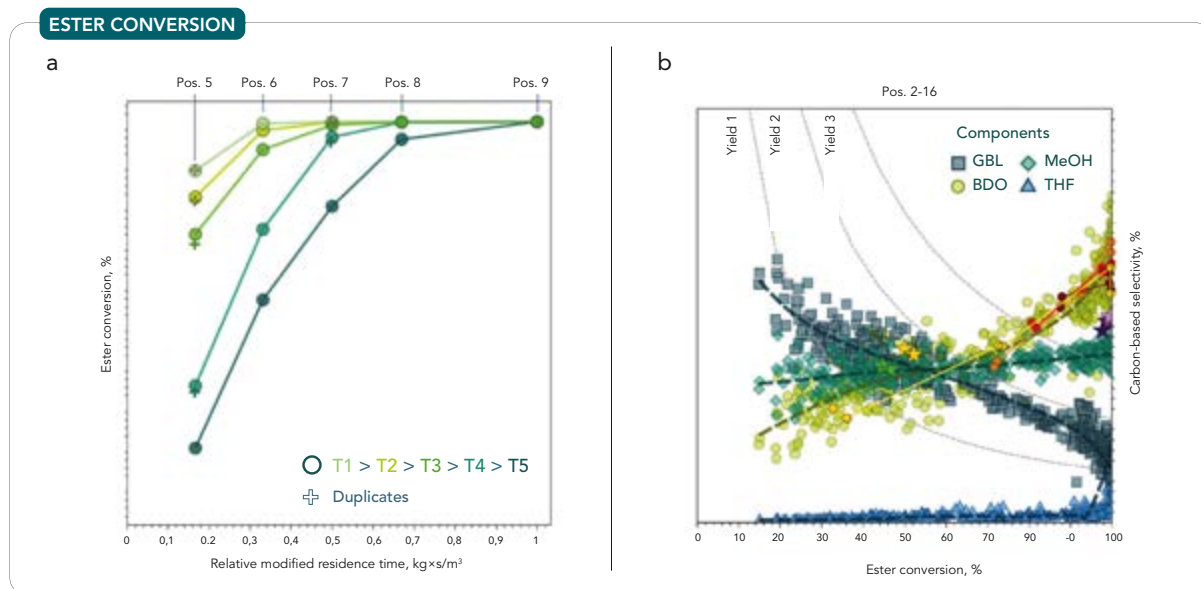


Figure 2. Graph (a) shows conversion versus modified residence time for selected reactors with constant guard-to-conversion-bed ratio at different reactor temperatures, while graph (b) plots carbon-based product selectivity versus ester conversion of bulk data with highlighted experiments at different reactor temperatures (red circles), and spatial catalyst arrangement (stars).

±1K). Nitrogen dilution of the reactor effluent reduced partial pressure of high-boiling products to avoid condensation. Safety condensers trapped heavy product components. The challenge posed by high volume flow was overcome by adapting the downstream assembly to reduce the pressure drop across the entire unit. A PolyArc gas-chromatograph (GC)/flame-ionization-detector system analyzed the product stream for enhanced oxygenate detection.

RESULTS

The experimental design exposed the 16 reactors to 16 different process conditions consecutively. This combination resulted in 256 experiments with more than 1,280 data points; these were screened within only 22 days during April and May 2020. As we will discuss, activity and selectivity profiles as well as decay data from these high-throughput experiments can support catalyst performance evaluation.

The combination of hot-gas flow meter and GC analysis data with a detailed assignment of all product species allows the closing of material (carbon, hydrogen) and flow balances across the entire rig.

The multistep hydrogenation of DMM to BDO is an exothermal reaction that proceeds with volume contraction. High pressure and low temperature thermodynamically favor high product yields. However, the desired parameter space resides fairly close to the dew point line of the main product and needs improved catalyst design on the one hand but very precise process control on the other hand to avoid condensation inside the pores of the catalyst.

Figure 1 shows how temperature and feed dilution were decreased until the dew point line was touched to verify the catalyst performance at this desired sweet spot. Precise control of temperature and partial pressure allowed the recording of data close to phase transition. Fluctuation of the mass balance increased near the expected condensation point of BDO. Beyond this critical condition, the carbon balance dropped significantly and liquids were recovered in the safety condenser. The results do not suggest a superimposed condensation based on catalyst pore effects, which is a desirable ability for these kinds of catalysts.

Facile filtering and clustering of the bulk data supported by the advanced data-handling capabilities of myhte gave quick access to the relevant correlations. For example, Figure 2a shows the conversion-versus-residence-time chart; it only considers reactor positions with equal guard-to-conversion-bed mass ratios.

The catalyst exhibits excellent activity. Full

conversion was achieved, even when operating far below a residence time of 500 kgxs/m³. Supply of extremely high flows by the rig decreased the residence time toward an area of differential conversion. Elevating the temperature increased ester conversion significantly. Raising partial pressure of the substrate had a similar effect. Duplicate positions, shown by cross symbols, reveal very good reproducibility of conversion and selectivity (not shown) over the entire conversion range.

Reaction network and performance analyses were carried out using superposition plots that allow for a tailored filtering of individual results within different layers. The background of Figure 2b shows the bulk data split into the selectivity of the main components versus ester conversion of all experiments. The dashed lines are provided only to better illustrate the selectivity curves. The target reaction is a multistage hydrogenation. DMM is converted to gamma butyrolactone (GBL) as an intermediate (squares) within the initial step, splitting off two methanol (MeOH) molecules as a couple product (diamonds) that will not be converted further. This can be shown by extrapolating the trajectory of the selectivities toward zero conversion. GBL and MeOH selectivity end up between 0 and 1. The selectivity of the BDO (circles) as the target product is zero at the initial step, suggesting it is formed consecutively from GBL. Running high conversion leads to dehydration either of GBL or BDO to form tetrahydrofuran (THF) (triangles).

A closer analysis of the product distribution revealed the formation of side products starting from both ester and the diol. Accordingly, feedstock losses will occur due to a parallel and consecutive side reaction. The reaction particularly is prone to forming polymeric products if

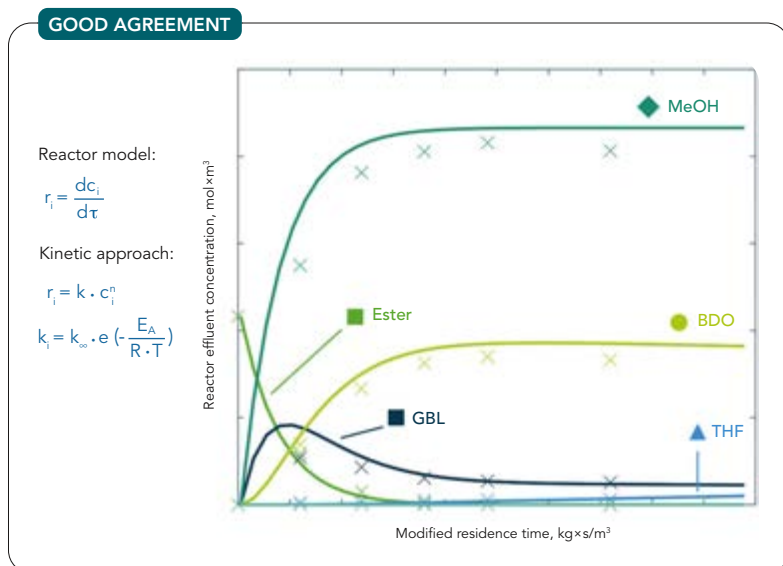


Figure 3. Model data (lines) quite well match measured results (crosses) for reactor effluent concentration versus modified residence time.

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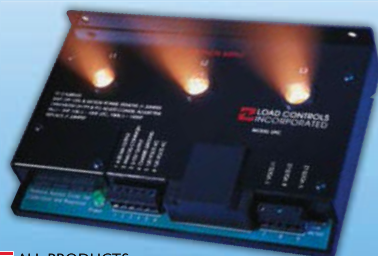
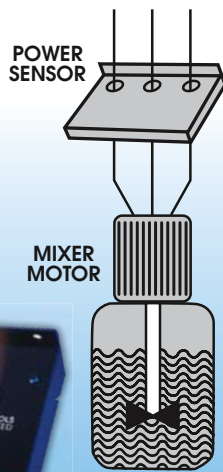
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operated at very high temperature and residence time. Therefore, reactor design and choice of operating parameters should take this into account.

However, within the tested parameter space, the side product yield was negligibly low, showing a good ability for selective diol production of the given catalyst. The overall yield (by carbon) of GBL, BDO and THF, which represent high-value commercial products, exceeded 70%.

In Figure 2b, yellow to red circles show the influence of reaction temperature on the BDO selectivity. In between, an ester conversion of 0–100% selectivity is independent of temperature. Running toward full ester conversion, the BDO yields meet the calculated values for thermodynamic equilibrium. At the highest temperature, a significant drop of the diol selectivity indicates the shift toward formation of the consecutive product (triangles) as well as increased side product formation.

Although the purpose of the guard bed (yellow star, Figure 2b) is to protect the downflow catalyst against impurities or initial side products, its target product selectivity is high and comparable to the main conversion bed (green star, Figure 2b). Accordingly, the commercial reactor will waste no space with a lower-performing layer. Purple star symbols indicate different catalyst ratios. High guard bed contents (dark symbols) and low guard bed contents (lighter-colored symbols) show product yields at the same level. This enables processing at a low content of top guard layer, which, in turn, significantly reduces the effort needed during catalyst top-layer skimming. Within the entire screening, the catalyst system showed good stability. The tested catalyst combines high activity and selectivity, leading to a superior space/time yield.

Fitting a formal kinetic approach to the experimental data enabled determining a preliminary mathematical description (Figure 3). It assumed isothermal conditions and plug-flow behavior, and neglected mass-transfer limitations. Hydrogen had been fed in excess and, therefore, is not considered within the equations. The balance for an ideal plug-flow reactor and power law approach were used as model equations. All data were fitted simultaneously to obtain the activation energy, frequency factor and reaction order of each single reaction pathway.

The calculated data are in good agreement with the screening results and allow for a prediction of effluent concentration as a function of temperature and residence time. Kinetic data models can support the experimental design during the implementation of new catalyst product lines and reduce downtime. This approach shows how



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high-throughput units can assist in determining precise kinetic data setups.

FOLLOW UP

The process was successfully transferred from commercial to laboratory scale; the application of high-throughput experimentation allowed for fast and efficient screening of activity, selectivity and decay behavior. Results agreed very well with the data from the commercial plant.

Based on hte's results, Sipchem intends to trial the catalyst at the commercial unit's next catalyst turnaround, which should occur before the end of this year. The results predict a longer runtime and overall yield enhancement of between 20% and 40% depending on the plant conditions. These benefits are of great value because they significantly raise the margin of the plant.

Sipchem and hte intend to work together on future projects. The current study provided a good understanding of the behavior of crushed catalyst processed with model feeds. Further work could involve testing shaped catalysts and processing real feedstocks. ●

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Put Soil Remediation on Solid Ground

Fixing fouling and other issues demands deeper digging

THIS MONTH'S PUZZLER

We've been operating a soil remediation process for about three months. It takes in chloro-compounds like polychlorinated biphenyls and dioxins. The solids-handling part of the process works flawlessly but biological fouling problems with the solvent extraction circuit (figure online at <https://bit.ly/3v9OMx9>) began to arise about two weeks after start-up.

We take in soil from places a hundred miles away or so. There's little quality control — we accept almost anything. However, our operators know to reject loads with logs, gravel, concrete and asphalt chunks. One truck delivered a stop sign! Our screw conveyors seem to handle what we take in.

We get our solvent, toluene, from pharmaceutical plants when they're not burning it in their thermal oxidizer. Originally, we planned to source it from refineries but we can't compete with the price the aviation industry pays.

We first noticed the problem in the vacuum systems drying the soil; then, it affected the flocculation in our settler and, finally, the extractor itself. I found a similar pond scum in our recycled solvent tank.

Other challenges for me are the rapid decrease in the quantity of recovered solvent as well as odor complaints from our neighbors in the industrial park. I think we may have to put in a scrubber system that we can feed back into our solvent recovery train.

Did we miss a treatment step? What could the residue be? What can be done about this?

SOLVE THE FOULANT PROBLEM

You should take a three-pronged approach. Consider:

1. control of foulants at the source;
2. foulant control in transport and in processing steps in equipment; and
3. anti-foulant treatment (e.g., dispersants, defoamers, biocides).

Because soil supply and soil sources are not monitored, it is hard to pinpoint the origin or origins of foulants. Broadly put, there are three major sources of foulants: inorganic materials (e.g., insoluble calcium salts, silica or other insoluble salts), organic compounds (e.g., grease, oil, wax, high-molecular-weight organics), and bio-foulants (e.g., plants, algae, small animals).

See if it is contractually possible (and practical) for you to require suppliers to check soil samples and provide you with analyses or certificates of analysis. You may restrict fouled soil.

Look into the suppliers and the way soil is delivered to you. Transport trucks carrying soil could also leave oil and grease. You could require cleaner trucks.

Check your process equipment (for oil, grease and other sources) and solvents to make sure they are free of foulants.

Options 2 and 3 may *not* be possible. You will need to consider use of anti-foulants and treatments.

Many anti-foulants, dispersants and defoamers are available on the market. Discuss your specific application with appropriate vendors. Along with cost effectiveness, consider environmental and safety issues.

*GC Shah, consultant
Houston*

WATCH YOUR BOTTOM LINE

The process has several problems; some are manageable, some are not. I don't even know if the process can be run economically, i.e., in the black.

First, there is the nature of toluene. When exposed to air in your process, the bugs that are present will gradually break down the toluene aerobically. The more oxygen that is present, the faster the bugs eat, leaving a sludge behind. The clue was the odor complaint: if toluene is getting out, air is getting in. Using pharmaceutical toluene also introduces additional nutrients that the aerobic bacteria will eat. It is nearly impossible to completely isolate a process like this one, so take steps to protect it from fouling.

You will want to look more closely at the filtration process. Consider adding a flocculant specific to bugs and a sock filter. Socks are good down to about 1 micron; they can be re-used and are fairly easy to maintain and troubleshoot. A more-expensive more-complex and less-reliable option would be a centrifuge.

Second, dioxins are C-Cl and C-Br compounds. These often are fatal to bugs, so the organisms will convert the compounds into ones they can easily release, like vapors. Unfortunately, those compounds are lethal to other organisms, like humans. If your state environmental protection agency hasn't caught on to this, you'd better start working on a plan to manage it: you can't hide the problem forever.

Which brings me to my third point: an odor scrubber. Putting in a vacuum scrubber is an excellent

Continued on p.44

Forestall Fouling-Factor Foul-ups

Accumulated errors can invalidate calculated results for heat exchangers

THE OVERALL heat-transfer rate ($U_{overall}$) achieved in a heat exchanger depends upon the overall resistance to heat transfer, which is its reciprocal ($1/U_{overall}$). This, for a unit with a wall between two streams, is the sum of five factors:

$$\frac{1}{U_{overall}} = \frac{1}{h_{outside}} + \text{foul}_{out} + \text{resistance}_{wall} + \frac{1}{h_{inside}} + \text{foul}_{in} \quad (1)$$

where $1/h$ is the film resistance for each surface, and the other factors are the resistance of the wall or tube, and the fouling resistance at each surface. Figure 1 shows these factors for a tube in a shell-and-tube heat exchanger. Many users have some sense of the fouling factor likely for most of their heat exchangers.

Of course, some services prompt more fouling than others. For example, a heat exchanger using steam usually has very little fouling on the steam condensing side. In contrast, the process side of a heat exchanger often may foul very heavily. In heat integration services with the process on both sides of the exchanger, fouling may occur on both sides at the same time. Experience also shows that apparently minor changes in exchanger configuration or process conditions can dramatically shift fouling rates from those generally expected for that service.

Direct measurement of fouling resistance is extremely difficult and expensive; it's rarely done even in research facilities. So, most fouling resistances come from calculations based on exchanger performance. Such calculations give overall fouling but don't break down each side's contribution. Experience may allow allocating how much fouling belongs to each side of the exchanger. Often, though, only an overall fouling resistance is stated.

Understanding fouling is important because our calculated fouling levels provide information for exchanger cleaning and maintenance decisions, attempts to mitigate fouling (e.g., additives and operating changes), and purchasing decisions. However, what really is our calculated fouling factor?

Simplifying Eq. 1 by taking the wall resistance as zero and combining the inside and outside fouling, we get:

$$\text{Fouling} = \frac{1}{U_{overall}} - \frac{1}{h_{outside}} - \frac{1}{h_{inside}} \quad (2)$$

The resultant value comes from subtracting two relatively large numbers from another large

number. This concept poses potential problems, as I've discussed previously (see February 2015 issue's "Resist the Temptation," <http://bit.ly/2HxY9kl>). When using plant data to determine the fouling factor, all errors in the calculation accumulate in the final result.

Plant data are one source of error. We measure the flow rate and the temperatures around the exchanger to calculate duty and provide information for the heat-transfer calculation methods. Are the flow rates and temperatures measured right? We also need the composition of the stream to estimate its physical properties such as viscosity and heat capacity. This becomes especially challenging in exchangers that are partial condensers or partial vaporizers because you really require the rate and composition of each phase.

The correlations we use are another source of error. How good are the correlations for the physical properties needed? In addition, how much scatter appears in the heat-transfer correlations themselves?

A recent analysis I did illustrates how these errors can accumulate. This in-depth study of 60 heat exchangers in multiple units indicated that five had a calculated negative fouling factor after cleaning when their units started up after a shutdown. Another eight had a calculated zero fouling factor.

These 60 exchangers had been in service for more than ten years and had gone through multiple shutdowns. They all received standard commercial handling and cleaning during the



What really is our calculated fouling factor?

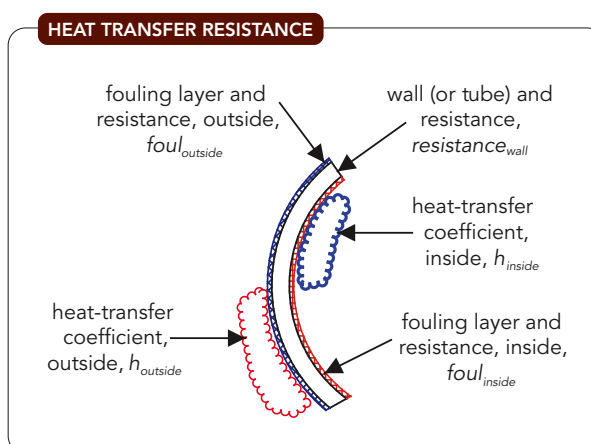


Figure 1. Tube in a shell-and-tube exchanger illustrates the five factors that contribute to the overall resistance.

shutdown maintenance. It's highly unlikely they were perfectly cleaned during the shutdown. Also, each one had some amount of corrosion and exchanger wear accumulated over the years. They surely had some fouling resistance present.

The calculated negative-to-zero fouling factors result from data-measurement and physical-property errors as well as correlation uncertainty. In this sample, at least 20% of the exchangers performed better than expected. Due to the physical condition of the exchangers, it's

difficult to say how many performed much worse than expected. Nevertheless, it clearly exceeded 20%.

Fouling factors are useful concepts for monitoring and identifying areas where a plant should take remedial action. However, don't blindly accept the calculated results — understand the possible errors and how they might affect your operating decisions. ●

ANDREW SLOLEY, Contributing Editor
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PROCESS PUZZLER

Continued from, p.42

idea but one fraught with its own set of challenges. There are four options: 1) a catalytic thermal oxidizer (TOX); 2) a bleach scrubber (60% removal); 3) a bio-trickle filter (89%); or 4) an activated-carbon filter. The least troublesome method is a TOX. The biggest challenge is providing sufficient fuel for the reaction. You could use natural gas to provide enough fuel to offset the air drawn in from the extractor or you could burn some of the toluene used as the solvent. In addition, the C-halogens require high temperature, perhaps beyond what you can get from gas, and scrubbers themselves for the products. Activated carbon works very well for lean toluene streams but the material only can be regenerated a few dozen times — then a TOX is needed

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anyway. Bio-filters work very well but are more hands-on than a TOX; again, there is the C-halogen problem with a very toxic sludge and vapor. However, a TOX does contribute carbon dioxide to the atmosphere. All of these issues affect the bottom line.

*Dirk Willard, consultant
Wooster, Ohio*

JANUARY'S PUZZLER

Our steam stripper isn't performing as designed. We are seeing 1,200 ppm of bottom contaminants instead of the 400 ppm expected. We anticipated foaming but it is much worse than foreseen, requiring us to cut back the tower rate. We tried raising the bottoms temperature and replaced our anti-foam pump with a larger one, increasing the addition rate.

The reduction in tower capacity, downstream purification of the bottoms and injection of anti-foam was killing our bottom line.

When we still couldn't meet necessary production and quality, we replaced the upper half of the sieve trays with two beds of random packing, a high-performance glass-filled polyethylene type. (We couldn't replace the sieve trays entirely because the temperature at the bottom of the tower exceeds the limit for the packing, i.e., 180°F.) We installed a distribution pan at the top of the packing

as well as a single vapor distributor in the bottom bed.

For a week after addition of the packing, the tower almost ran within specifications and we were able to increase capacity by 50% over the initial rate. I think we solved our foaming problem with a 50% reduction in defoamer addition; now, the pump is oversized.

Did we miss a treatment step? What could the bottom contaminants be and what can we do about them?

Send us your comments, suggestions or solutions for this question by December 10, 2021. We'll include as many of them as possible in the January 2022 issue and all on ChemicalProcessing.com. Send visuals — a sketch is fine. E-mail us at ProcessPuzzler@putman.net or mail to Process Puzzler, *Chemical Processing*, 1501 E. Woodfield Rd., Suite 400N, Schaumburg, IL 60173. Fax: (630) 467-1120. Please include your name, title, location and company affiliation in the response.



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The Maximum-Duty Aerolock rotary valve is engineered for heavy to severe industrial service with pressure differentials up to 1.4 barg (20 psig) and temperatures of 121°C (250°F). The unit is designed to withstand severe shock loads and elevated temperatures consistent with continuous 24-hr use. A ten-blade rotor maintains a minimum three-blade labyrinth seal to minimize air leakage. The rotor is available with flat or relieved tips in several rotor choices including open-ended, hubbed, reduced displacement and staggered pocket. The valve utilizes two tapered roller bearings on both the thrust and drive sides. Materials of construction include carbon and stainless steel with multiple coating options.



Coperion K-Tron

785-825-3830

www.coperion.com

Real-Time Sensors Enhance Feed Control

The Model 408-170-0 weight-loss weigh feeder has a compact 18- x 24-in. footprint and a metering mechanism designed to accurately and reliably meter a variety of dry solids. The unit's weighing system is permanently calibrated and adjustment-free. The Ratiometric Digital Weight Resolver performs high-resolution weight sensing to produce an unamplified, non-integrated real-time weight signal for precise feed control using one of the company's multiprocessor controllers. Designed with a flat-bottom feed chamber, the metering mechanism is housed in the chamber and comprised of a horizontally rotating conditioning agitator with a metering auger positioned underneath for highly effective and efficient feeding of materials.

Acriston, Inc.
201-440-8300
www.acriston.com



Emissions Monitoring System Simplifies Compliance

The Rosemount XE10 continuous emissions monitoring system is designed to help industrial plants meet increasingly stringent environmental regulations and evolving sustainability demands. With its built-in performance analytics and automated validation capabilities,

the system ensures accurate emissions monitoring and reporting to help plant operators reduce the risk of non-compliance shutdowns and penalties. Engineered for rapid deployment, the compact and standardized system is certified to the European standards EN 14181 and EN 15267, which govern the quality assurance requirements for automated measurement systems of

stationary source emissions. The system also complies with the emissions reporting requirements of the U.S. Environmental Protection Agency.

Emerson
888-889-9170
www.emerson.com/RosemountXE10

Software Supports Single-Site Operators

AnteaSTART mechanical integrity software is tailored to the basic needs of single-site operators. The software provides real-time visualization of assets, enabling operators to make quick, evidence-based decisions for improved



maintenance and reliability, reduced inspection costs and fewer shutdowns. It also includes data management support, which will save operators substantial time on data entry and reduce the likelihood of human error, says the company. It includes Antea Core, the base of the company's asset integrity management platform, as well as risk-based inspection, inspection data management, and electrical and instrumentation modules, with the option to scale up functionality as needs increase.

Antea
888-642-6832
www.antea.tech

Metering Pump Delivers Pulse-Free Flow

Hydra-Cell Metering Solutions P700 Series metering pumps feature a hydraulically balanced, multiple-diaphragm design to provide smooth, linear, virtually pulse-free flow without the need for expensive pulsation dampeners. Variable-frequency-drive electronic flow control adjustment pumps flow throughout the turndown range at up to 1,394 gph (4,396 lph) with discharge



pressure ratings to 1,200 psi (83 bar). Diaphragm position control protects the pumps and enables operation in case of a closed inlet. Because the process fluid isn't used for lubrication, the pumps can run dry indefinitely. According to the company, a replenishment valve in every piston assembly ensures optimum actuating oil on every stroke for continuous accuracy.

Wanner Engineering, Inc.
612-332-5681
www.hydra-cell.com/metering/product/P700.html

Mobile Bag Dump System Minimizes Dust

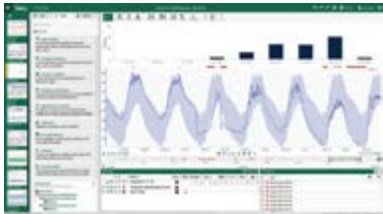
A sanitary mobile tilt-down flexible screw conveyor with integral bag dump station and compactor allows the transfer of material manually dumped from handheld bags into elevated process equipment, and the disposal of



empty bags, dust-free. Mounted on a mobile frame with locking casters and a fold-down step, the bag dump station is secured to the floor hopper with quick-release clamps. A gasketed bag disposal chute through the side wall of the hopper hood allows the operator to pass empty bags directly into the bag compactor. Dust generated from both dumping and compaction is drawn onto the system's two cartridge filters.

Flexicon Corporation
888-353-9426
www.flexicon.com





Software Expands Machine Learning Capabilities

R52 advanced analytics software includes new features to support the use of machine learning in process manufacturing operations. These features enable organizations to deploy their own or third-party machine learning algorithms into the advanced analytics applications used by front-line process engineers and subject matter experts, thus scaling the efforts of a single data scientist to many front-line OT employees. New capabilities include add-on tools, display panels, and user-defined functions for faster development and deployment of easy-to-use algorithms and visualizations for process engineers. End users also can schedule Seeq Data Lab notebooks to run in the background.

Seeq

206-801-9339

www.seeq.com

On-site Generator Reduces Oxygen Costs

The OGP+ on-site compressor and oxygen generation system eliminates the costs and logistics of buying, moving and storing the gas. By optimizing pressure swing adsorption (PSA) technology, the 8-model range reportedly consumes 30% less feed air at full load to produce the same amount of oxygen as a traditional generator, reducing the total cost per unit of oxygen. The unit also saves energy at low load with variable cycle saver



(VCS) technology. VCS optimizes the PSA cycle to reduce the air requirement to what is needed to generate a lower volume of oxygen. By eliminating energy waste during lower demand, VCS generates up to 70% extra energy savings, the company says.

Atlas Copco Compressors

866-546-3588

www.atlascopco.com/air-usa



Enclosed Drive Endures Harsh Environments

The Sinamics G120XE enclosed drive system helps meet the demands of industrial pump, fan and compressor applications. The system reportedly is ideal for fast design and commissioning in harsh industrial environments. A NEMA 1 enclosure is standard, with a NEMA 12 version optional, featuring appropriate ventilation and air filters. The base enclosure suits wall-mounting to 75-hp applications, while the free-standing floor module accommodates uses to 200 hp. Standard electrical characteristics include a UL508A listing, SCCR rating to 65kA, a circuit breaker disconnect with flange-mounted operator handle and mechanical door interlock, plus control power transformer for internal power control and power module with PWM IGBT inverter.

Siemens

800-743-6367

usa.siemens.com/sinamics-g120xe

Oxygen Deficiency Monitor Extends Sensor Life

The Alpha Omega Instruments Series 1300 oxygen safety monitor offers flexibility and improved user-configurable options. Its advanced digital interface permits expanding the unit with as

many as three oxygen sensors; a local sensor and up to two remote sensors.

Its extended-life electrochemical oxygen sensors are designed with an enhanced electrolyte system that increases sensor life expectancy. The digitally controlled instrument with a measuring range of 0–30% is housed in a resilient polycarbonate, wall-mountable general-purpose enclosure. Oxygen values are displayed to the nearest 0.1% on a high-contrast front-panel liquid crystal display. Also included are four individually adjustable Form C alarm relays.



Alpha Omega Instruments

713-947-9591

aoi-corp.com

Calculator Helps Improve Energy Efficiency

Determine the potential savings variable frequency drives (VFDs) can bring to chemical operations using this online energy savings calculator (<https://bit.ly/3lYhTR9>). The calculator shows potential energy and carbon emissions savings specific to each electrically driven asset. Users answer a few questions about



their application and the calculator will estimate the savings they can realize by switching to VFDs. The calculator reportedly suits applications that involve electric motors not currently controlled by VFDs. By using VFDs, companies can improve motor control performance and motor efficiency, and as a result lower their operating costs and improve their environmental performance, the company says.

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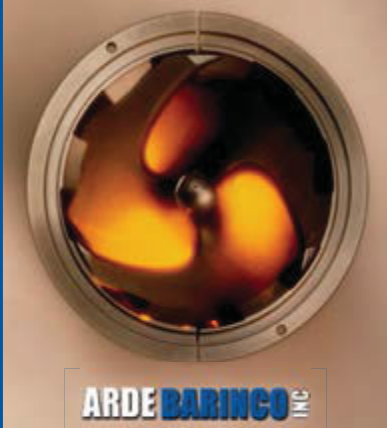
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Statement of Ownership, Management, and Circulation

1. Publication Title: Chemical Processing
2. Publication Number: 101-980
3. Filing Date: 10/01/2021
4. Issue Frequency: Monthly
5. Number of Issues Published Annually: 12
6. Annual Subscription Price: \$68.00
7. Complete Mailing Address of Known Office of Publication: 1501 E. Woodfield Rd. Ste. 400N, Schaumburg, IL 60173-6053
8. Complete Mailing Address of Headquarters or General Business Office of Publisher:
1501 E. Woodfield Rd. Ste. 400N, Schaumburg, IL 60173-6053
9. Full Name and Complete Mailing Address of Publisher, Editor, and Managing Editor:
 Publisher: Brian Marz, 1501 E. Woodfield Rd. Ste. 400N, Schaumburg, IL 60173-6053
 Editor: Mark Rosenzweig, 1501 E. Woodfield Rd. Ste. 400N, Schaumburg, IL 60173-6053
 Managing Editor: Amanda Joshi, 1501 E. Woodfield Rd. Ste. 400N, Schaumburg, IL 60173-6053
10. Owner: Putman Media, Inc., 1501 E. Woodfield Rd. Ste. 400N, Schaumburg, IL 60173-6053
 John M. Cappelletti, 1501 E. Woodfield Rd. Ste. 400N, Schaumburg, IL 60173-6053
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11. Known bondholders, mortgagees, and other security holders owning or holding 1 percent or more of total amount of bonds, mortgagees, or other securities. If none, check box None
12. Tax Status (For completion by nonprofit organizations authorized to mail at nonprofit rates.) The purpose, function, and nonprofit status of this organization and the exempt status for federal income tax purposes:
 - Has Not Changed During Preceding 12 Months
13. Publication Title: Chemical Processing
14. Issue Date for Circulation Data Below: September 2021



PUBLISHING HEADQUARTERS

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15. Extent and Nature of Circulation:		Average No. Copies Each Issue During Preceding 12 Months	No. Copies of Single Issue Published Nearest to Filing Date
a. Total number of copies (net press run)		30,957	30,924
b. Paid and/or requested circulation	(1) Paid/requested outside-county mail subscriptions stated on form 3541.	24,541	24,343
	(2) Paid in-county subscriptions stated on form 3541		
	(3) Sales through dealers and carriers, street vendors, counter sales, and other non-usps paid distribution		
	(4) Other classes mailed through the usps		
c. Total paid and/or requested circulation		24,541	24,343
d. Free distribution by mail (samples, complimentary, and other free)	(1) Outside-county as stated on form 3541	5,981	6,157
	(2) In-county as stated on form 3541		
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	(4) Copies Distributed Outside the Mail (Include Pickup stands, Trade shows, Showrooms and Other Sources)	21	0
e. Total Non-requested Distribution		6,002	6,157
f. Total Distribution		30,542	30,500
g. Copies Not Distributed		414	424
h. Total		30,957	30,924
i. Percent Paid and/or Requested Circulation		80%	80%
16. Electronic Copy Circulation:			
a. Requested and Paid Electronic Copies		18,000	18,000
b. Total Requested and Paid Print Copies (Line 15c) + Requested/Paid Electronic Copies		42,541	42,343
c. Total Requested Copy Distribution (Line 15f) + Requested/Paid Electronic Copies (Line 16a)		48,542	48,500
d. Percent Paid and/or Requested Circulation (Both Print & Electronic Copies)(16b divided by 16c)(100)		87.6%	87.3%

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17. Publication of Statement of Ownership. Publication required. Will be printed in the November 2021 issue of this publication.
18. Signature and Title of Editor, Publisher, Business Manager, or Owner: Brian Marz, Publisher, Date: 09/28/21
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3D-Printing Process Holds 4D Possibilities

Hybrid method combines manufacturing and chemical treatment to strengthen parts



The geometrical changes were retained.

RESEARCHERS AT Loughborough University (LU), Loughborough, U.K., have developed a hybrid additive manufacturing process to selectively control the mechanical properties of 3D-printed parts.

The process uses acetone to toughen targeted layers of 3D-printed parts and structures. The acetone, depending on the amount used and where it's applied, enables creation of novel mechanical characteristics, such as controlling how parts deform.

An article in *Additive Manufacturing* describes the new process, which is dubbed Material Treatment Extrusion Additive Manufacturing (MaTrEx-AM).

Essentially, the researchers added another property to products made by the established Material Extrusion Additive Manufacturing (MEAM) 3D-printing process. MEAM gives users the ability to rapidly manufacture custom parts with complex geometries. These, point out the authors, often are prohibitively costly or time consuming to achieve by traditional subtractive manufacturing methods.

MEAM operates by extruding molten polymer filament and depositing it via a heated nozzle onto a print platform, where it rapidly solidifies. Typically, the nozzle moves in the X and Y planes (parallel to the print platform) to generate layers of parts sequentially. The lowering of the print platform in the Z direction at completion of each layer enables depositing subsequent layers, leading to a layer-wise production.

Herein, note the authors, lies the biggest limitation in MEAM: mechanical anisotropy, particularly weakness in the Z direction.

Historically, this anisotropy has been attributed to deficiencies in the interlayer bonding, the researchers note. However, their research points to the true cause of interlayer weakness being geometrical, with naturally occurring filament-scale grooves causing stress concentration and reducing a load-bearing area by narrowing the bond region.

While chemical treatments have addressed this in the past, most focused on using acetone to improve surface finish of 3D-printed parts — with only the short-term effects of its use investigated. Furthermore, these studies did not analyze the evolution of mechanical and geometrical properties over time.

In contrast, the LU team maintain this is the first study to demonstrate the selective tailoring of mechanical and geometrical properties of polylactide (PLA) and acrylonitrile butadiene styrene (ABS) additively manufactured with material extrusion using

a newly devised treatment method of chemical immersion in acetone. This hybrid manufacturing and chemical treatment process is capable of modifying mechanical behavior in a predictable and time-controlled manner.

Analyzing short-term effects (tested immediately after immersion) generated important findings on mechanical and geometrical properties of printed specimens. Different immersion times (1–60 seconds) caused significant increases in strain-at-fracture — up to 25- and 16-fold for PLA and ABS, respectively. The researchers found, in the long-term, mechanical properties recovered by up to 90% for untreated specimens in ABS within three hours, while PLA properties recovered after 60 days.

Importantly, while mechanical properties in both PLA and ABS recovered, the geometrical changes were retained. Annealing allowed for shorter recovery time: PLA specimens fully recovered within 24 hours.

“Chemical immersion provides significant advantages over widely used vapor-based treatment methods, since it allows selective application to be achieved and localized changes of mechanical and geometrical properties,” they write. So MaTrEx-AM enabled increased toughness and a redirection of strain into selected regions of the mesh material.

Andy Gleadall, senior lecturer in additive manufacturing in LU's school of mechanical, electrical and manufacturing engineering, says, “The process adds material layer-by-layer — there are grooves between the layers, a bit like you'd see if you stacked lot of logs sideways on top of one another, all lined up. 3D-printed parts are often weak because of the way layers are laid down in sequence, so there are geometric defects between the layers and the bonding of material between layers may not be as good as the pure polymer.”

“The new capabilities are potentially valuable for a huge range of parts and structures, but perhaps most obvious ones would be parts that deform during operation and the way in which they deform needs to be controlled. The time-dependent nature of mechanical properties means the approach adds a new dimension to material capabilities, with *in-situ* hybrid processing facilitating a true 4D-printing process,” he concludes. ●

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