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PILOT PLANTS

PILOT PLANTS: DESTINED FOR DEVELOPMENT

NEW IDEAS HATCH IN PROCESS DEVELOPMENT

OUTSOURCING INNOVATION DEVELOPMENT

PILOT PLANT FABRICATION: INSPIRATION TO OPERATION GUIDANCE

PILOT PLANT CORNER

PILOT PLANTS: DESTINED FOR DEVELOPMENT

Pilot plants are on the verge of an unprecedented evolution. Read about the 10 factors that'll impact the design, construction and operation of these next-generation units.

I have seen many changes in pilot plants over the course of my career, but I predict that we are on the verge of an unprecedented evolution of these units. My crystal ball sees 10 key factors influencing next-generation pilot plants:

- outsourcing;
- automation;
- fugitive emissions;
- multiple trains;
- online analytical capabilities;
- safety and control system interaction;
- wireless technology;
- instrument availability;
- instrument multi-functionality; and
- unit size.

Let's look at each of these and what they may spur.

Outsourcing

Contractors will play an expanding role in supplementing or replacing in-house resources in the conventional design, construction, start up

and operation sequence — prompted by companies' desires to be more efficient and responsive while minimizing commitments to longer term in-house resources. This will range from contract design, construction and maintenance to increasing use of outsourced analytical services, programming and even operations. The greater flexibility contracted services offer to gear up for a sudden short-term need or scale back during an industry downturn will prove irresistible to many organizations. However, those firms apt to be the most successful will maintain some in-house expertise — at a reduced level overall but concentrated in more depth and considered more of a strategic resource. Companies will continue to value in-house design skills but will be more willing to bet that an outside firm can design a pilot-plant vessel right or built it just the way they want. The most successful will recognize the need to maintain some fairly high level of expertise to find, evaluate, review and select the best contractor — and probably to do some or all of the unit process design — as well as to make use of the resultant pilot plant and its data.

Automation

Manual operation has already almost completely given way for operation of all but the simplest pilot plants. Automation currently is moving along the path of reducing operating staff attendance from essentially full-time down to progressively less-and-less part-time. The next generation of units will require even lower operator presence and make much greater use of recipe-driven menus that allow the operator to select the operations sequence from a master list and then depart, secure in the knowledge that the pilot plant will properly execute each step (well, at least most of the time). Different pilot plants will employ the same sequences, as organizations strive to develop a more-standardized approach to common opera-

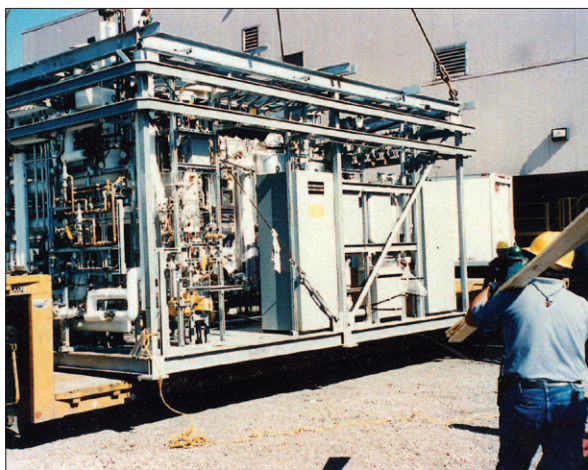


Figure 1. This pilot plant, built at one site and is being shipped to another site of the same organization, exemplifies “in-house” contracting.

tions like charging, pretreatment and sampling. Efforts to develop the “best” approach will make these operating sequences more uniform and sharable. Examples include more-complex charging, filling and preparation arrangements, automated sampling protocols and even operational sequences like planned experimentation based on the latest test results.

Fugitive emissions

Increased toxicity of materials, reduced exposure limits and growing concerns for the long-term health effects of any exposure will push efforts to design and construct units that are leak-free under all circumstances. Decreasing operator attendance, which reduces the time available for identifying and locating leaks, also will promote this trend. The combined health and operational concerns will spur companies to install more equipment that is less leak-prone. Sealless pumps and mixers, bellows-seal valves, and high-integrity fittings typify the leak-free components rapidly becoming common on pilot plants. Automatic tube welders, which make welding easier and a more viable alternative to conventional joining methods, will proliferate, while specialty closures and assemblies will increasingly replace conventional flanges and piping. More and more instrumentation will come as sealed units or with higher-integrity seals. Routine automatic online leak detection, currently rare and intermittent, will become more popular to address the reduced operator presence and ensure safety when no one is around.

Multiple trains

The reduced staffing that automation makes possible, coupled with the enormous expansion in data work-up and mining capabilities offered by today’s computers will promote the increased use of multiple trains. This will increase the complexity of pilot plants as well as their support and maintenance requirements — but the added

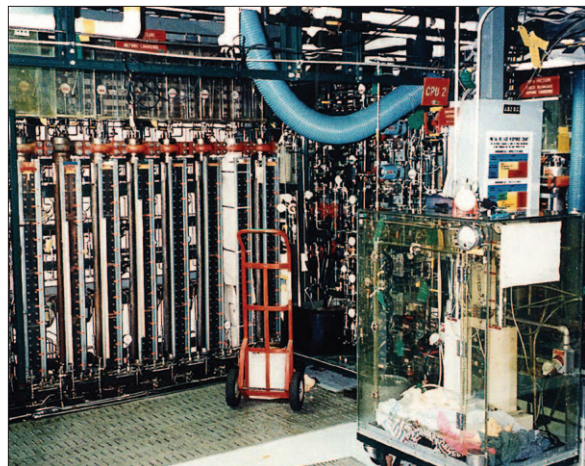


Figure 2. A highly automated elastomer pilot plant replaced several smaller units that required more manual operation.

productivity and effectiveness will outweigh the higher costs. Such setups may consist of multiple trains on the same unit or multiple copies of a single unit, depending upon the organization’s requirements. They will provide not only traditional data but also more-in-depth analytical and operational results for use in evaluation and design.

Online analytical capabilities

Over the last 20 years, the number of online analytical tools has dramatically grown. This trend will accelerate as analytical data become more integrated into process operation and not just data analysis. Process control based on real-time analytical data, already increasingly popular, will widely spread. Process optimization, only just beginning to grow in pilot plants, will proliferate. More importantly, integrating these data into the pilot plant’s control system will become more uniform and hence easier and less expensive. Third-party programs, integrated systems and common bus structures will allow the data to be fed to the process control system in a more-straightforward, less-proprietary



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manner. The complexity of the pilot-plant control system will grow as these inputs are integrated to the maximum feasible extent. More-difficult analyses such as particle-size distribution and complex product compositions will gain a greater role.

Safety and control system interaction

The age-old separation of control and safety systems has largely blurred into being almost unrecognizable on many pilot plants. Growing concern over how well the safety system will respond should the control system be unavailable or non-functional will force pilot-plant



Figure 3. Large, heavily automated group of distillation pilot plants provides major savings in design and construction as well as more efficient operation.

control systems in new directions. In some cases, simplified layer-of-protection analysis will lead to an overall safe design. In others, the prevalence of separate microprocessors in a single control system will allow operation safely in both modes, given proper initial configuration. Continued use of separate control and safety systems will remain common for the foreseeable future, but they both will be microprocessor-based, smaller, cheaper and more failsafe, as well as easier to integrate and program. Integrated systems that have separate microprocessors on each board or rack will become more common and provide the redundancy a safety system requires.

Wireless technology

We have only begun to scratch the surface of using wireless technology for pilot-plant operations. While the long distances between sensors and control, which are driving this technology in plants, usually do not exist in pilot plants, its lower cost, greater flexibility and reduced construction time make the technology too attractive to ignore for much longer. As wireless devices become cheaper and more common, thanks to their use in plants, they will gain greater acceptance for pilot plants. As time goes on, they will replace the usual hardwired systems from small tank farms and remote operations. We will see greater use of wireless highways not just to gather data but also to transmit data to end users and storage.

Instrument availability

Small-scale magnetic flow meters, vortex meters, corrosion probes and numerous other devices were but a dream for most pilot-plant designers 20 years ago. Now many are becoming increasingly common and low cost. The growth in this area will continue. The availability of these devices will allow pilot-plant designers to solve some issues that have plagued them for years (much as the advent of thermal mass-flow meters in the 1970s finally put to rest the search for an ultra-small-size control valve to use with differential pressure devices). The resultant boost in accuracy and reliability will, in turn, enable pilot plants to produce valid useful data with every run — obviating statistical analysis of several runs to address inaccuracy and non-repeatability.

Instrument multi-functionality

Multi-functional units will proliferate. Pressure transmitters will simultaneously measure temperatures; flow meters also will provide pressure or density in a single unit. Calibration of most new transmitters will occur while the unit is in place and online. While the individual transmitter will be more expensive, it will be smaller, more accurate and more reliable. The decrease in installation costs will more than offset the higher purchase price. These multi-functional units also will inter-

face more easily with control and data-acquisition systems, generating additional savings in programming and set-up.

Unit size

The days of the size of pilot plants shrinking every generation are probably approaching a realistic end. However, the use of very small high-throughput “pilot plants” (which actually are more akin to very complex experimental equipment) will increase. These high-throughput units will handle much of the screening currently performed more slowly and expensively in standard small pilot plants. Highly automated pilot plants then will run the promising leads at a more realistic and scalable range, to evaluate synergistic effects and operations at transient conditions as well as process conditions more realistic of a plant environment. The combination, when properly applied, will produce a greater number of high quality leads faster, and provide a means to screen these for the next generation of process or product improvements. Modeling will continue to augment and validate pilot-plant operations and, in the always symbiotic relationship, pilot plants will continue to augment and validate modeling.

Cost impact

The combination of all of the trends described will translate into increased cost to design, construct, start up and operate next-generation pilot

plants. It also will raise the expense and effort to keep these pilot plants effectively running. The days of maintenance support being a few craft-people on loan from the plant or hired when needed through a local contractor are over — although many maintenance functions will be routinely outsourced for cost or to gain access to specialists. Just as the “tooth to tail” ratio in the modern military keeps getting smaller as the lethality of weaponry and their associated complexity increase, so the “unit to support” costs of pilot plants will shrink; the data will become better, more useful and more focused — but keeping units working properly will incur higher costs and effort. The traditional process and mechanical engineering support requirements will continue, matched now by computing, automation, safety and electronics support requirements.

Will all these predictions come to pass? Probably not, although I think most will, in some form or another. Beyond these, I forecast that an even-more-novel trend, not mentioned nor even imagined by most pilot-plant personnel, will arise and significantly change the way we all design, construct and operate our pilot plants. After all, that’s what research is all about, change, both planned and predicted, as well as new and unexpected!

These predictions, of course, represent my personal view, not necessarily that of Exxon-Mobil or any of its affiliates.



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NEW IDEAS HATCH IN PROCESS DEVELOPMENT

Don't expect pilot plants to disappear as better tools and techniques enhance efforts

Compared with the rapid pace of product introductions elsewhere, the chemical industry can at times appear to lack innovation. As a mature and necessarily conservative industry, it certainly cannot be expected to match the pace of change in, say, consumer electronics and mobile telephony. But appearances can be deceptive. All across all the chemical industry, from the realm of high volume, low margin bulk petrochemicals to high-added-value fine chemicals and pharmaceuticals, new products, and the processes to make them, are constantly under development, albeit more slowly and more methodically than in some other industries, where perhaps fashion is a more important driver than function.

Bipin Vora, senior corporate fellow for process technology development at UOP, Des Plaines, Ill., speaking at July's World Congress of Chemical Engineering (WCCE) in Glasgow, Scotland, put it this way: "Successful technology development, from concept to commercialization, requires a structured process. It may take anywhere from five to 10 years, requiring substantial expenditures in terms of research, pilot plant construction, development, scale-up, engineering design, and economic analyses."

Benchmarking

Central to process development in most cases is the pilot plant, although in reality it is but one link in a chain that starts on the laboratory bench and ends at the plant with a fully commercialized unit. At the WCCE, results of an American Institute of Chemical Engineers (AIChE) pilot-plant benchmarking study were presented for the first time to an open audience. Thirty companies from across the commodity chemical, specialty chemical, pharmaceutical and oil and gas industries in North America took part in the exercise, a three-year project completed by the association's process development division toward the end of last year.

The reasons why companies decide to pilot new and improved processes vary across the industry sectors, noted David Edwards of Zeton, Burlington, Ont., chairman of the division's pilot plant group. Overall, they pilot either to demonstrate the viability of new processes, to generate design data or to produce market development samples of product. Different sectors have different priorities, of course. Sample production scores high among the pharmaceutical companies, for example. It is far less important to the oil and gas sector, which relies on piloting primarily to prove the viability of a new process and generate reliable design data.

Likewise, sectors showed distinct differences in how they decide which potential processes should progress through to the pilot stage. Approaches include: opting to pilot all processes, using a formalized risk-assessment process, making the choice based on an informal team or individual judgment, conducting a systematic review process, or relying on "stage gates" in which specific stages of the development process have to be completed before moving on to the next step.

The only sector that prefers to use piloting for

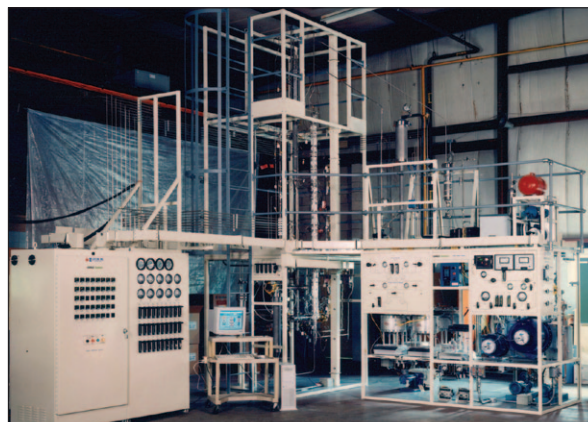


Figure 1. A pilot plant remains central to process development, for evaluating processes, generating data or making sample quantities of product. Source: Zeton Inc.

all process development is pharmaceuticals, with 57% of respondents from that sector saying their company chose this route. The bulk commodity chemical industry, on the other hand, overwhelmingly appears to take the view that the decision to pilot should be based on a formal risk assessment of the process concerned.

Dan Pintar, operations manager at UOP's Riverside, Ill., facility and a member of the AIChE team conducting the benchmarking survey, says the "gated" approach used by his company is a good way of involving multidisciplinary teams in the development process at an early stage. "Chemists might establish 'proof of principle' from their lab work," he explains, "but to get to the next stage of development you have to pass through a stage gate, which is when you get input from the process development engineers." And it's the engineers, along with representatives from the commercial side of the business, who give the "thumbs up or down" to allow the project to move to the next stage.

According to Vora, at this and later stages of the process development, "the statistical design of experiments can and should play an important role, to better understand the results and minimize redundant efforts." These experiments are likely to be on a small bench scale, often with the aim of screening various catalyst formulations or determining the range of operating parameters. Vora sounds a note of caution here, however. "Because a bench-scale 'pilot plant' often does not have product recovery or internal recycle streams built in," he says, "the results need to be taken with a certain level of healthy skepticism. The results achieved under a perfectly controlled environment may not translate as well, or at all, to real-life situations."

It's at the next stage, the actual pilot plant, that issues such as the impact of the various recycle streams and impurity buildups can be fully assessed. "Even though the commercial design of the project may be several years away," Vora says, "input from all the various branches of engineering design is critical at this early stage."

High stakes

UOP focuses on developing processes to license and, so, is understandably cautious because it actually will not be running the processes. Some operating companies that do their own process development also see merit in a measured approach. With facilities in Houston, Texas, and Amsterdam in the Netherlands, Shell Chemicals' Chemical Process and Development Group has delivered many new processes and process improvements to the company's operating sites around the world. Heading the process engineering and evaluation group in Houston is David Torres, who says: "It's important to learn early on that an idea has merit before millions are spent building a unit." His group does preliminary process design and economic analysis to guide research programs and to determine the economic viability of a project.

As to whether companies would prefer not to pilot at all — relying instead, for instance, on process simulation models developed from lab-based experimentation — the AIChE survey (which, after all, was of pilot plant users) probably doesn't provide many answers. But Zeton's Edwards says that, while companies may be more selective about which processes they pilot in the future as resources become scarcer, "the need to pilot at a meaningful scale before moving to a commercial scale will always be a requirement. The risk is too great in not piloting a new process, because there are always surprises during piloting — byproduct accumulation, catalyst performance issues, corrosion issues

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and so on — and models are not too good in revealing such surprises (Figure 1)."

Acknowledging that his views are those of someone working for a company that designs and builds pilot plants for others to operate, Edwards nevertheless acknowledges the importance of the other stages of process development. "I think lab work, computer models and pilot plants all have an important role to play in process development," he says. "Fundamental lab work will always be needed and a model can screen which potential new processes should be piloted."

UOP's Pintar takes a similar view. "There's always going to be a need for the pilot plant in our current paradigm," he argues, "because people want to see data. They want to see proof. Although, if you could develop a good kinetic model based on your pilot-plant data, then you might not need to run the plant all the time to generate estimates for customers or to do revamp studies. The problem is that we are always trying to push the units, to push the processing conditions outside of the regime for which the model was built."

Faster screening

To develop any process model, however, presupposes a process in the first place. And for this we still need the laboratory bench and what was once the laborious work of screening many different compounds and assessing how they react under different catalytic conditions. This is now the realm of combinatorial chemistry — in which large numbers of reactions can be performed simultaneously in high throughput, small-scale systems.

An example is the HTS (High Throughput Screening) system of Symyx Technologies, Santa Clara, Calif., which has just won Frost & Sullivan's 2005 Technology Leadership Award. "Symyx's high throughput approaches offer significant advantages over conventional methods of catalysts discovery," says F&S industry analyst, Anil Naidu. "The systems can rapidly screen materials to achieve the desired properties, delivering results faster and at a much lower cost."

The success of Symyx's combination of high throughput experimentation with its propri-

etary software tools for handling the data produced was highlighted last year with the start-up by Dow Chemical in Tarragona, Spain, of its first commercial plant to produce Versify plastomers and elastomers. These speciality propylene-ethylene copolymers are manufactured using a new catalyst system developed in collaboration between Dow and Symyx. "This is an important milestone for Symyx," commented chairman and CEO Steve Goldby, "when an innovative discovery coming out of our labs goes into full commercial production."

Earlier this year, Symyx announced a \$120-million five-year strategic alliance with Dow "to effect a broad change in Dow's R&D capabilities and efficiencies." This deal follows a similar alliance with ExxonMobil signed in 2003 to run for five years and worth more than \$200 million to Symyx.

As noted earlier, companies in the pharmaceuticals sector tend to have different priorities in process development than the bulk commodity chemical producers. According to David Ainsworth of engineering/procurement/construction contractor Foster Wheeler Energy, Reading, U.K., the use of simulation models — such as Batch Plus from Aspen Technology, Cambridge, Mass., and SuperPro Designer from Intelligen, Scotch Plains, N.J. — can help pharmaceutical companies investigate numerous design alternatives quickly and easily. "The computer model adds value at all stages of the design process," he says, "from early conceptual design through to the ultimate operation of the facility."

Ainsworth also cites the value that early involvement of an experienced process contractor can add — particularly in the pharmaceutical industry where processes are typically developed by teams of chemists. "Analyzed in a methodical manner, the specific characteristics of each process become evident and alternative processing methods can then be identified."

Better tools

In its analysis arsenal, Foster Wheeler includes weapons developed by Britest, Cheadle, U.K. This not-for-profit company was set up in 1998

by a group of leading chemical and pharmaceutical companies, including AstraZeneca, Avecia, GlaxoSmithKline and Rhodia, to follow up on new approaches to process technology coming out of the universities at the time and to encourage technology transfer. Using what are known as the Britest tools — a set of proprietary procedures and software programs — is, says Ainsworth, a time-effective way of starting the development process and determining all the potential (and infeasible) process options.

One area the Britest toolkit considers is process intensification (PI), not just in the development stages but through to the commercial stage, as well. At Zeton, Edwards also is seeing a trend among the company's pilot plant customers towards PI techniques and equipment — “although it's still very much in its infancy,” he says. “We think operating companies are going to be interested enough to want to try it, but nervous enough not to go full scale until they have tried it on a pilot scale.”

UOP's Pintar notes that at the same time as “pilot plants themselves have shrunk in terms of reactor size,” there is a growing drive for more data collection and on-line analyses from the plants. Fulfilling both of these goals, a new process analytical tool has recently been successfully trialed by specialty chemicals producer Clariant Chemicals at its plant in Leeds, U.K.

The plant used a patented “constant flux” reaction calorimeter developed by Ashe Morris, Radlett, U.K. (Figure 2). The Coflux technology — which is akin to a variable area, rather than variable temperature, heat exchanger — is said by co-developer Robert Ashe to permit stirred tank reactors of virtually any size or type to be operated as precision calorimeters, offering a simple solution for on-line monitoring of chemical and biological processes. The R&D manager at the Leeds plant, Jim Wilson, said Clariant was able to monitor the rate of change (powder



Figure 2. This “constant flux” reaction calorimeter recently got its first plant use at Clariant Chemicals' Leeds, U.K., site. Source: Ashe Morris

dissolution and reaction) throughout the trial experiments and could successfully detect the start and finish of each step in real time.

Real-time monitoring and increased automation were certainly among the trends identified by the AIChE study, as was an increasing emphasis on the safety of pilot plant operations. This highlighted something of a paradox because, as Pintar observes, despite the increasing levels of automation, “the majority of companies don't allow unattended operation of their pilot plants.”

No doubt this will be a topic for the next benchmarking exercise, expected in three to five years time. Until then, however, the future for the pilot plant at the heart of process development seems assured.

OUTSOURCING INNOVATION DEVELOPMENT

A new development method may allow breaking away from the pack as companies continue to employ outsource facilities that offer flexibility and foster innovation.

Innovation, whether through the development of new products or processes, has become crucial for companies in virtually every industry. New technology holds the key to competitive advantage and, perhaps, survival. Product life seems to be shorter and shorter (Figure 1). For manufacturers, the problem is particularly acute because of the ease with which competitors can outsource production. No longer can mature companies, with established manufacturing bases, count on an economy-of-scale advantage as a barrier to entry from new competitors.

Since 1964, Pressure Chemical Co. has worked with hundreds of customers from the large multinational corporations to small entrepreneurial start-ups, applying unique configurations of equipment and extensive chemical expertise. These efforts have often resulted in new products, processes and, at times, new business segments for the client company. As expected of developmental projects, however, many failed to produce a successful innovation, though, in retrospect, a large number of the attempts did yield substantial savings for the sponsoring client. Early “failures” have prevented a company from making large investments in a process that wouldn’t work as anticipated or in a product that couldn’t meet the performance and economic needs of the marketplace.

“We’ve been able to observe scores of successes, near misses, and failures – the entire range of potential results,” says Larry Rosen, CEO of Pressure Chemical. “In an effort to improve our own internal processes, we began to examine the data closely and realized that we had learned to do things in a new and different way, having seen the best and the worst of all the organizational processes used by our customers.” With the help of an outside consultant, Droz and Associates, we cataloged our new products and processes that illustrated

a variety of circumstances, parameters and goals. The consultant found that our method was a radical departure from traditional budget-driven, stage-gate approaches (Figure 2).

Although attempts have been made to improve the traditional method, such as the Critical Path Method (CPM), a joint venture between DuPont and the Rand Corp., the approach was flawed. The Japanese, perhaps driven by their respect for W. Edwards Deming, evolved a step-gate method that looks back periodically to correct the trajectory of a project. Still, something was lacking.

Droz helped us to conceptualize the approach in a graphic manner that illustrates the central distinction of this cyclic route from the traditional straight-line approach. The key benefits of this novel approach, which we call Concept to Commercialization (C2C), are reduction in time, cost and risk, akin to a hat trick in hockey, according to Rosen.

The goal of a recent project, undertaken for a major international manufacturer, was the hydrogenation of a polymer for use in high-capacity

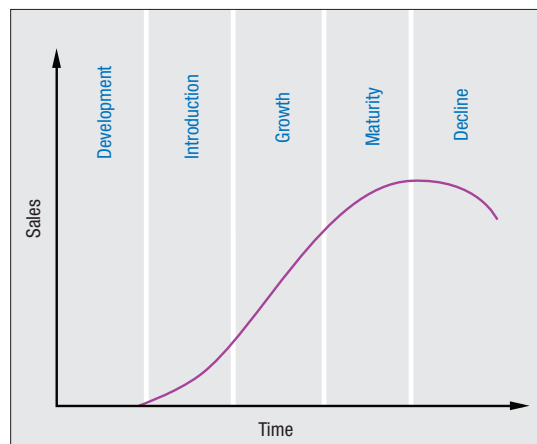


Figure 1. The lifecycle of a product — to stay in business a company needs a steady stream of new ideas.

data storage. This client chose to outsource the project because of the diversity of appropriately sized equipment available in our facility. The base polymer had been produced by the client in its large continuous production facility and the scope of work was limited to hydrogenation. Unfortunately, market testing of the target product revealed that its properties failed to meet expectations. Because it wasn't feasible to interrupt commercial production to produce small quantities of differentiated precursors for further work, the client faced abandonment of the project.

In discussions centered upon future windows of opportunity to process additional samples, the client was introduced to the variety of resources and interdisciplinary team of specialists that could be assembled to move the project forward without substantial delay. The proposal presented to the client expanded the scope of work to include creation of a small polymerization system and synthesis of the triblock copolymer precursor. Within three weeks, the project was back on track and demonstrating the best features of the nascent C2C Method.

The C2C method

The C2C method has a number of key distinctions: Unlike traditional, linear models of product development, it's a cyclical process where one cycle inputs into the next and where a variety of solutions move repeatedly through a

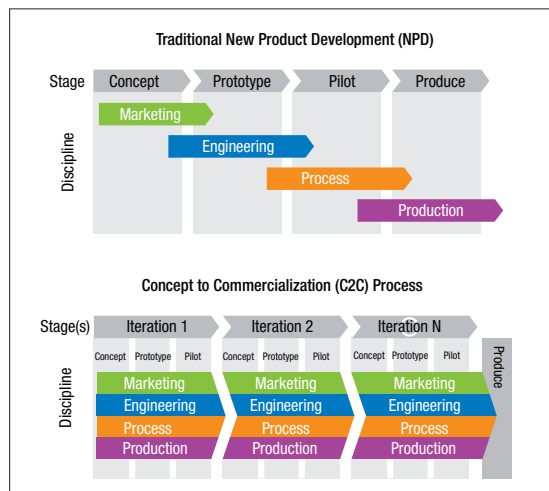


Figure 2. The cyclic C2C approach can correct the trajectory of a product during its development.

range of stages. It integrates rapid prototyping and multidisciplinary teams to allow numerous, and nearly simultaneous, iterations. Inspired, in part, by approaches and techniques commonly employed in food industry test kitchens, this method requires a devoted team, incorporating all appropriate disciplines and allowing a broad range of process options for comparison and contrast as to efficacy, scaling and suitability.

This method typically postpones confirmation of a concept until several iterative cycles have been conducted, to preserve flexibility and to allow incorporation of new ideas into a synthesized



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set of solutions. Traditional approaches frequently focus early on a preferred outcome rather than permitting the open consideration of alternatives.

In C2C, numerous potential processes may be evaluated and ranked for strengths and weaknesses. Experimental work and iterative prototype testing determines the right combination of conditions for each potential stage or step in the process. By combining unit processes that are most promising, a new process train can be defined, installed and tested, incorporating the best attributes and practices of the variations considered. And, of course, as with tasting in a “test kitchen,” the product is sampled, analyzed and tested without delay.

Why outsource development?

Companies outsource work for many reasons, often expecting to reduce costs and time to completion or to resolve resource availability issues. Sometimes the reason is safety, secrecy or anticipated production problems (Figure 3). Many companies presume that cost is the easiest factor to assess and, consequently, they allow the purchasing department to evaluate the decision to “make or buy” developmental services. Unfortunately, many purchasing executives lack the information for an in-depth analysis and understanding of all relevant costs and risks. For example, in comparing the “price” quoted by an independent facility to an internal “budget,” a purchasing executive may ignore critical risk factors or competition for internal resources simply because that information is not presented to him.

Some companies have saved millions of dollars by employing outsourced facilities to take the risks in scale-up, notable among them, firms in the pharmaceutical industry. There are several examples in our database where the world’s foremost experts in a particularly narrow field of chemistry learned to their horror that the impossible does occur!

In one memorable case, a client company assured us that its fluorinated product was entirely stable and couldn’t damage our all-glass, high-vacuum distillation system (Figure 4). The glassware was replaced. Had this work been performed in the client’s facility, the notoriety and delays in

incident investigation and equipment replacement might have had disastrous consequences for other products and work scheduled in their facility!

“There are so many constraints for companies — some initially unforeseen — in new product development,” says Mike Keenan, a retired senior chemist from Exxon who has worked and consulted on a number of projects at Pressure Chemical. “Since many companies are committed to existing technologies, it’s difficult for them to have the equipment, capital and, sometimes, the mindset to develop new products and processes efficiently. And companies vary in their strengths. Some are superb at taking someone else’s process and making it more efficient and effective. Others are better at discovering a new process from scratch. In any event, outsourcing certain stages of the product development process can bolster total development efforts,” according to Keenan.

“You need to develop new products outside of the typical constraints of manufacturing, preferably where you can brainstorm for ideas with operators, chemists, mechanics, engineers and regulatory specialists,” Keenan added. “You need to be in a place where change is anticipated and facilitated, not where change requires sign-off at several levels and can take weeks or months.”

Changing equipment and process procedures are germane to the development process. “Unanticipated issues arise during scale-up; it’s common to change equipment and conditions midway through the development process, even during the course of a reaction” said Brandon Ritchie, a senior project manager at Pressure Chemical. “It’s much easier to change something in a well equipped pilot plant than in a client’s production facility (Figure 5). Safety, flexibility and speed are everything in process development,” he added. Pressure Chemical’s project leaders are given full authority to accept client initiated changes in equipment and operating conditions so long as the change conforms to defined safety requirements.

For example, a new client project required some dramatic modifications to the distillation of a high melting monomer. The attempted distillation resulted in a lot of freezing in the process piping. The problem was solved by injecting an

appropriate solvent into the overhead to deliver the product as a solution. “We had the ability to modify the equipment quickly and to develop a new, highly successful process for the distillation,” Ritchie said, adding that this preserved the delivery schedule for the product.

Regulatory issues

Large companies are well aware of the impact of federal, state and local regulatory issues in product and process development. Smaller companies, especially ones that do not manufacture novel chemical products, may be totally unaware of the regulations affecting new chemicals. An independent pilot facility that specializes in innovative materials maintains an awareness and working knowledge of the rules, limitations and regulations impacting its customers’ development efforts. For those without the internal regulatory capability, an early consultation with an independent pilot facility should at least identify regulatory issues.

Companies will often base their new product specifications on their lab scale work with research-grade reagent chemicals in the lab. These self-imposed, tentative standards may not be feasible on a commercial scale but, frequently, provisional as they may be, these specifications take on the weight of authority and nobody remembers why. A major component of the innovation process, applicable to new chemicals, is the appropriate product specification and the techniques by which they are to be measured. Unnecessarily tight specifications may limit the market because of excessive costs while inappropriate specifications may allow a process to be scaled-up and commercialized before it’s ready.

A recent example of tentative specifications drawn too tightly comes from the development of a process to manufacture a novel cosmetics ingredient. The original lab work, performed in 100-ml. lab glassware, employed high purity reagent chemicals and produced a high purity product after high-temperature distillation. Unfortunately, a slow but steady decomposition at the necessary distillation temperature produced a highly undesirable and irritating byproduct. By changing the stoichiometry of the synthesis, using an excess of a reagent

commonly employed in formulations that would include this product, the distillation step could be eliminated, increasing the yield and reducing cost.

Because the tentative specification had been prematurely communicated in product literature, the client was forced to delay acceptance of a change until its customers had agreed. Not only was the client saddled with the associated higher costs, but it was unable to meet the initial demand for its new product.

“If you gear your process to making a high purity product, you’ve got to ask yourself: ‘What is the cost to meet this level of purity?’ Sometimes it is best if the question is deferred and the answer postponed to the end of the development process,” Ritchie said.

A flexible alternative

The traditional straight line, stage-gate approach to development has been the industry standard for many years. We believe the innovation process can be enhanced by using a cyclical process where multiple solutions, shepherded by a multidisciplinary team, move through the development stages. Outsourcing offers a flexibility that is essential to introducing new ideas, throughout the development process — creating a rich synthesis of solutions. By outsourcing work to the appropriate facility, companies will find that they can achieve a reduction in the time to market and the risk of failure while realizing a lower “real” cost of development. To learn more about product development and the Concept to Commercialization (C2C) process, visit www.pressurechemical.com.



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PILOT PLANT FABRICATION: INSPIRATION TO OPERATION GUIDANCE

Eliminating surprises in the process

Introduction

Bringing new products to market quickly is a business imperative in virtually every industry. A methodical approach that minimizes process and product quality risks in developing new technologies is also critical. To that end, pilot plants play an essential role in transforming R&D concepts into commercially viable processes for chemicals, catalysts, fuels and other products.

Although the overall design-build-test-operate cycle for pilot plants follows similar steps for most technologies, these can be unfamiliar and even daunting to those who haven't been through the process before.

This article provides insight into the pilot plant development cycle, with the goal of understanding the process for companies that are seeking a design and fabrication services provider to help them develop and commercialize technologies. This article assumes that the company has established proof-of-concept at the bench scale and is now ready to scale-up.

Scope

Pilot plants are necessary to minimize risk encountered when developing new and unproven technologies. The distinguishing characteristic of a pilot plant is that its main "product" usually is data – not a large volume of physical goods. These data include engineering design information for the commercial plant, impact of operating parameters on process efficiency and product quality, feedstock qualification, development of safe operating procedures, and assessment of capital and operating costs. Consequently, pilot plants must be more robust and flexible than their commercial manufacturing counterparts.

Pilot plants are built to withstand extremes in process conditions, since optimal manufacturing temperatures, pressures and other elements of the process have not yet been established.

What this means for the pilot plant "owner" is that the scope of the project is more extensive than if the unit was built only for physical goods production. Economies of scale, extra instrumentation, more flexible process configuration and conservative construction philosophy all lead to relatively higher costs for these development units. Many organizations that have never designed, built or operated this type of custom equipment often underestimate the cost of the unit, even though they recognize the importance and value of their pilot plant. The goal is to find the balance between building a pilot plant that is sufficiently large and flexible, yet is no more complex and costly than required to meet your objectives.

The fabricator's engineers can work with you to assess the purpose, size, operational flexibility, and the extent of automation and analytical equipment required to meet your project goals. A fabricator's experience, ingenuity and understanding of the chemistry that is the foundation of the process being tested can make a big difference in this regard. These competencies, as well as commercialization experience, are advantageous to have at hand when implementing a vital tool for advancing your technology.

Engineering Study

Before the first component is bought or weld is made, an engineering study should be done. Determining the depth of the study is a first step in this part of the project. The more detailed

the study, the more accurate the cost estimate for the pilot plant will be. A fairly cursory, preliminary study can yield a +/- 50% “budget estimate,” which can help you make a go/no-go decision for the project, or decide to commit funds for a deeper study. The more extensive study, which can narrow the cost variance to about 10%, will enable appropriate allocation of funds and ensure that all the parties involved – the owner, fabricator and any subcontractors – understand their role, the schedule and the deliverables.

A thorough study calls for significant effort on the part of the designer/fabricator. It is not uncommon for the engineering study to represent up to 10% of the total project cost. The benefits of investing in this effort include lowering project risk and costs through: tightening the project scope, identifying all major equipment, projecting labor costs, enacting preliminary safety and operations reviews, and developing a critical path schedule. This study provides the information necessary to make sound decisions about the pilot plant project and, consequently, the pace of technology development.

A prime consideration in the design and operation of the pilot plant is safety. Because the unit will likely be used to define operational limits during the process of technology development, the design needs to incorporate redundant safety systems and eventually enable the overall system to “fail safely.” Operator safety is of the utmost importance, and means going beyond the normal practice of addressing safety regulations relevant to the facility where the unit will operate and any industry regulatory mandates.

Keep in mind that it’s not only the conditions inside the processing system that matter. Units that are being fabricated in the process of scaling for “real world” systems that

might run in the heat of the Texas summer or cold of the Canadian winter call for considering the effect of extreme environments. That will enable the service provider to determine a parts list with components that can tolerate the all operating conditions and to build a pilot plant that will more effectively provide the data and product you need to assess and refine your process. By utilizing all the tools available, relying on your fabricator’s experience, and anticipating your needs in the “real world” system, you can advance toward commercialization and minimize the need for changing the system during fabrication.

Detailed Engineering

Once the scope of the project is finalized and the engineering study is complete, the project is ready for detailed engineering and fabrication. Detailed engineering involves sizing and specifying equipment (including materials of construction, instrumentation, service requirements, etc.), creating detailed process designs, and identifying the precise layout of the equipment. This is a major part of the process. At this stage, the owner’s involvement is often reduced, since the major part of the effort at this stage is for the engineers to turn the scope and results of the engineering study into final specifications and drawings.

A detailed engineering study provides everything needed to fabricate, including:

- Project schedule
- Process flow diagrams
- Block flow diagrams
- Calculations
- Process & instrumentation drawings (P&IDs)
- Specifications and drawings for all equipment (process, mechanical, struc-



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- tural, piping, electrical and instrument)
- Control philosophy and automation
- Safety systems

Fabrication

With all the essential, up-front engineering finished, fabrication can begin. At this stage in the process, it is best to not introduce change orders. Change orders are generated when deviations from the scope of work are required during fabrication – which even includes removing components from the system. Considering that most projects are meticulously planned as a step-by-step series of tasks with deadlines, it's easy to understand how modifications significant enough to warrant a change order will increase cost and time of fabrication.

During fabrication, documentation on the system and its operation are created. Final documentation includes:

- As-built version of all the review documents
- P&IDs
- Equipment layout
- Process bill of materials including instruments (specifying tag, manufacturer, model, basic size or connection, basic materials, design pressure/temperature, calibration, special notes)
 - Equipment data sheets
 - PSVs (pressure safety valves)
 - Designer notes
 - Equipment vendor technical and maintenance manuals
 - Material certifications
 - Pressure vessel certifications

Multiple copies of the documentation are usually provided and frequently include electronic copies.

Inspection & Testing

During fabrication, individual components will be tested for functionality and their ability to meet operational conditions, for example leak checking of pressurized systems. But,

ultimately, the complete system needs to go through a shakedown. Inert gases and simulating fluids can be used to run the unit at a series of conditions, so that the system is tested at the flows, pressures and temperatures that the unit will see during normal operation.

Several layers of testing are important during checkout. First, the unit should be tested for completeness, meaning that all the process, electrical, instrumental and computer materials and components that were included in the bill of materials and design are present, meet their specifications and are properly tagged. The system needs to be tested for mechanical functionality and leaks; pumps, valves and other components also undergo a functional test to verify the performance of the equipment and associated instruments. All the controls also will be tested during the functional test. Finally, all safety systems must be activated to ensure that the unit “fails safely.”

Operation

Once the pilot plant is fabricated and tested, the next step is operation. There are three options for operations:

1. Delivery of the pilot plant to the owner's site for installation and start-up
2. Brief operation by a contractor before delivery
3. Long-term operation by a contractor

Option 1 is most typical. Most owners want to start operations themselves, as they have already operated the process at smaller scale and may have proprietary procedures or chemicals that they want to maintain as secret.

Options 2 and 3 can be extremely valuable to the customer. Experienced operators can run the pilot plant for the customer, both to ensure that the unit is operating as expected, under “real world” conditions, and to optimize the process. The fabricator can train the owner's operators during this time as well, while using collected data to

determine if final equipment changes are required. Using the fabricator as operator also can be valuable if the owner does not want to increase staff, or has limited experience with pilot plant operations.

Summary and Conclusions

While new methods for simulating the behavior of complex processes have been and continue to be developed, nothing can substitute for the knowledge gained through actual operations. Just like commercialization, pilot plant fabrication and operation is a step-by-step procedure that calls for both experience and creativity. The pilot plant is a critical vehicle for technology development, a tool to help ensure that your innovation enables your company to succeed.

About Continental Technologies

Continental Technologies designs, builds, installs and can operate processing equipment for technology development and scale-up. The company specializes in pilot plants for fuels, catalysts, chemicals, polymers and solids development and production.

About the Authors

Doug Jack, VP of Technology at Continental Technologies, has nearly three decades of experience in energy and petrochemicals with

a primary focus on the development and implementation of technology for commercial applications.

Len Dolhert, VP of Business Development at Continental Technologies, has over 20 years of technology commercialization experience in the areas of equipment, chemistry, electronics components and advanced materials.



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Questions, Answers and Recommendations

Pilot plant digester mixing

Q: I would like to continually mix 4% DS anaerobic sludge in a 1cu.m pilot plant digester(h/d aspect ratio 1.3/1), using an external pump system with 2 nozzle injections. Sludge throughput is 3l/hour. @ 37degC. Max. solids size = 10mm. What would be minimum injection velocity and flow rate required? Any other requirements i.e nozzle angles, nozzle, injection height, baffles, etc.?

A: The answers to all of these questions appears to be the reason for the Pilot Plant Digester. If someone could easily answer these questions, the pilot plant would not be needed. The pilot plant digester should be designed and constructed in a manner such that the nozzle diameter and pump rate can be changed easily to test what the minimum injection velocity and flow rate is. The nozzles must be easily relocated to test the effects of nozzle angle, injection height, location, etc. Baffles may be necessary to avoid bypassing of the main digester flow.

The real question is how do you scale-up results from the pilot plant size to the full-scale digester. The answer to the scale-up question, must also come from the pilot plant. Tests need to establish whether maintaining jet velocity is sufficient to maintain performance or whether flow rate has a more significant effect. Particle size and size distribution may have an effect on the answer to the scale-up question since concentration is a factor on both flow and particle suspension.

<http://www.chemicalprocessing.com/experts/answers/2008/041.html>

Creating a pilot plant for a continuous process

Q: We have a process wherein precipitation occurs when reagent A (a 70% aqueous solution) is added to reagent B (a 30% aqueous solution). Precipitation is instantaneous and a batch of

6,000 kilograms of active ingredient is completed in 90 minutes. It takes 480 minutes to filter the slurry through a batch filter, and we wish expand the process threefold. How do we convert this process into a continuous one? We can install a straight-line filter, but how do we set up a pilot plant to optimize the continuous process?

A: The pilot plant likely will not have as full of a process train (not as much equipment) as the full scale process. For the pilot plant, consider two tanks in series for the precipitation, with the second tank agitated and feeding a pilot-size drum filter. If a drum filter is not available, filter cake tests will have to be performed on stationary filter media with measurement of the volume or weight of slurry per filtration area vs. time. Optimization of the filtration parameters may be desired. The filtration rates (in lph/m² or gpm/ft²) can be scaled-up by the multiple of the of the full scale process compared to the pilot scale process.

<http://www.chemicalprocessing.com/experts/answers/2006/020.html>

Recommendation for small pilot plant batch mixer

Q: I'm looking for a recommendation for small pilot plant batch mixer (ideal capacity target 1 to 4 liters) that handles powder and can be heated to about 80 C. Mixing should be possible at low speed.

A: The size is at or below the typical size for laboratory powder blenders and the temperature is at the high end of the operating range. Some liquid blending equipment is capable of handling free-flowing powders and may be suitable. The powder characteristics are very important, since mixer type depends on powder flow and processing requirements. Custom or modified standard equipment may

be needed to meet the requirements.

<http://www.chemicalprocessing.com/experts/answers/2006/013.html>

Formula for position of impeller

Q: We have a pilot mixing plant here on site. Ever since it was stripped down and rebuilt due to its PM schedule, it hasn't worked properly. We use the tank to mix a powder and a liquid. Now when it runs, no vortex is created (or be it a very poor one) and the powder does not go into solution, it just sits on the top of the liquid. We have tried moving the impeller up and down the shaft but to no avail. Unfortunately, the original dimension drawing are missing and so we do not know the original position of the impeller and its dimensions etc. Are there formula that might help us to determine the optimum position for the impeller etc. if we measure the various aspects of the tank, baffles etc.?

A: Powder incorporation is almost art as it is science. From the information given, PM should not have directly caused the problem. Possible solutions:

1. Increase the mixer speed - that may have changed with maintenance.
2. Increase the liquid level, so it extends above the top of the baffles, depends on the tank straight side and baffle length.
3. Reduce the distance between the impeller and the liquid surface, this dimension has the greatest geometric effect on surface motion and vortex depth.
4. Type of impeller - pitched-blade works best as upper impeller.
5. Use multiple impellers - two impellers will increase surface motion and maintain mixing.

The problem is well understood, the correction is unusual, as described the process worked before.

Recent literature on pilot plants

Q: Can you assist me in recent literature on pilot plants (books or papers)?

A: A good overview can be found in both the Kirk-Othmer Encyclopedia of the Chemical Process Industries and Ullman's Encyclopedia.

You can also look for the following books:
Pilot Plant Design, Construction, and Operation

Richard Palluzi
McGraw-Hill, 1992
ISBN 0-07-048180-6

Pilot Plant and Laboratory Safety
Richard Palluzi
McGraw-Hill, 1994
ISBN 0-07-048181-4

There are also some of my articles available:
Improving Pilot Plants (Chemical Engineering, June, 2001)

Cost Effective Pilot Plant Design and Construction (Chemical Engineering, April, 2000)

Succeed at Crash Pilot Plant Construction (Chemical Engineering Progress, Dec, 1997)

Choosing The Right Pilot Plant (Chemical Engineering Progress, Jan, 1991)

Pilot Plants (Chemical Engineering, March, 1990)

AICHE also runs three courses on Pilot Plants (#192, 193 and 244). They can check the AIChE website (www.aiche.org) for details.

Difference between pilot plants and mini plants

Q: In Germany we distinguish between pilotplants and miniplants. What is your definition?

A: In the US the term "miniplants" is rarely used. More commonly some version of "Lab scale" or "hood size" pilot plants is more common. Alternate terms include "lab units" or "Lab scale pilot plants". We just don't seem to make that much of a distinction. Another example of common experiences separated by a common language!

About the Sponsor

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Continental Technologies fabricates processing equipment used in development and scale-up of technologies including chemicals, fuels, catalysts, polymers and solids. The company also can operate the equipment it builds, offering an additional option to those who cannot or choose not to operate, which enables speeding time to operation and saving money while also getting the data and product critical to development. We build from the end user's perspective, producing modular systems that are easy to use and adapt while never losing sight of safety.

Whether you are looking for proof-of-concept at benchscale, scale-up operations to pilot-plant scale, need to test your process across a range of operating conditions, demonstrate commercial viability at large scale or enhance your chemical processing operations, Continental Technologies can help.

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