

POLYMER PROCESSES TAKE FLIGHT WITH PILOT PLANTS

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In today's competitive polymer industry, manufacturers must find new ways to develop high-quality, value-added products and bring them to market before the competition does. New and existing products, with added properties, are competing in established commodity and specialty markets, while new manufacturing processes continue to be developed.

ELK GROVE VILLAGE, ILL—Increasingly, petrochemical companies use pilot plants to improve polymerization activities. Although their most basic use is in catalyst research, where small batch reactors aid in screening and developing new catalysts, larger continuous polymerization pilot plants are essential to develop new products and processes.

Pilot plants for developing catalyst manufacturing processes are also essential for new technologies.

Specialty polymer pilot plants are perhaps most beneficial in process and product research and development. Automated pilot systems can simulate commercial polymerization processes and determine the viability of new products. They help reduce process uncertainty, improve product quality, decrease product loss and increase throughput.

Essential to the design of a polymer pilot plant is a computer-based control system—such as the Honeywell PlantScape system or, as is the case at this Xytel installation, the Honeywell Scan 3000/Series 9000 system—including data acquisition, process control, safety logic and data manipulation. Microprocessor-based control systems and programmable logic controllers are most useful for this purpose. An array of smart pressure transmitters, temperature transmitters, single-loop controllers, analyzers and other devices monitor key process performance indicators and enable the plant to perform a host of automated tests. Use of microprocessor-based, digital technology in these systems offers improved data integrity and reliability and eliminates problems associated with operator bias and data interpretation.

Types of plants

Computer-controlled pilot plants and demonstration units are used with a host of polymerization technologies, including gas, bulk, condensation, emulsion, solution and slurry phases. Polymer pilot plants range from small units for kinetic studies and catalyst screening to large development plants with throughput ranges between 10 kg per hour and 200,000 pounds per year.

Various pilot plants and process demonstration units can be constructed with new, modular systems, such as those developed by Xytel Corp. of Elk Grove Village, Ill. Pilot plants can be divided into three types: process development, product/market development and technical service.

Process development plants

Process development pilot plants produce data that is valuable in commercial plant design and operation. A typical process development application for a pilot plant might involve duplicating a reaction scheme—such as two loop reactors followed by a fluidized gas phase reactor in a series—or modeling a downstream process, such as centrifuging, drying, or extruding. Other common applications include testing the effects of feed impurities, catalyst variations, process variables, heat transfer and mixing variations.

For a new or modified process, scale-up is a critical factor. Particular attention must be paid to the selection, size, and scalability of critical equipment. Here, too, pilot plants can provide useful information that will aid in the development of commercial units.

Typically, process development pilot plants are equipped with instruments that provide heat/mass balance around critical unit operations. Such pilot plants can run in adiabatic or isothermal modes, depending upon the design of the commercial plant. They generally include recycle streams that are a common source of commercial plant problems.

Process development pilot systems are rarely at locations other than a commercial plant site because their infrastructure makes it impractical for them to be at research and development facilities. Process development plants frequently evolve into product/market development and technical service pilot plants. Product development and technical service studies are often accomplished in the same research pilot plant.

Product/market development plants

One of the most important uses (and economic justifications) for pilot plants in polymerization is in product development. The recent trend for polyolefin producers has been toward market differentiation. Manufacturers are more inclined to develop value-added products than commodity products. Product/market development pilot plants are used to facilitate this process.

Located in close proximity to a commercial plant, product/market development pilot plants are designed to repeatably produce product for client and in-house evaluation purposes. The pilot plant's capacity is based on product evaluation techniques and market introduction strategies. Recycle streams are normally included so they can significantly impact product capability.

Product/market development pilot plants are growing in use because of the significant drawbacks to trying out untested catalysts or new grades in a commercial plant. Aside

from lost production of high-value polymer during changeovers and unsuccessful test runs, product development in a commercial unit often results in the production of large quantities of off-spec or low-value product that must be disposed of, and possible shutdowns because of plugging or operating problems.

Product development in a commercial environment is also inhibited by the limited availability of on-stream research time, the difficulty of scheduling research runs, and interruptions to production operators' normal work schedule. The fear of these and other major problems limits the creativity and scope of researchers' efforts.

Technical service plants

A technical service pilot plant is designed to troubleshoot operating problems in a commercial plant. Theories can be accurately tested and strategies safely implemented in the pilot plant before attempting them in the commercial unit. Such problems as plugging, poor selectivity, low product quality, and erratic control can be investigated in the pilot plant environment.

The configuration of a technical service pilot plant is made easier by the availability of design data from an existing commercial unit. For this reason, the pilot plant is normally situated at the commercial plant site.

Technical service pilot plants are also a valuable asset for plant operator training. Training on a pilot plant that is similar in design to the commercial unit offers minimal risk and maximum interaction with the process.

Catalyst applications

Two of the most common applications for pilot plants in polymerization are catalyst screening and evaluation.

Catalyst screening. In polymerization, catalyst screening is intended to investigate kinetics, product properties and activity. The screening is used to identify, from a large number of catalyst formulations, the most promising catalyst systems in terms of key performance indicators. Pilot plants enable existing commercial or newly generated laboratory samples of catalyst to be compared.

A small-capacity catalyst screening system is necessary where laboratory-generated catalyst samples are to be investigated. If the capacity (throughput) of the catalyst screening system is too large, then multiple samples may be needed

Catalyst screening pilot plants must also remain small to allow for rapid start up, stabilization, data collection, and shut-down cycle times. The throughput is designed to only be large enough to provide product samples for melt index and other simple rheology tests.

Pilot plants used for catalyst screening often perform the actual polymerization in a different mode than the technology which the equipment is supporting because the results needed in this phase of study are qualitative in nature. In other words, a fluid bed gas phase polyolefin technology may be supported with a catalyst screening system, whereas

the polymerization is carried out in a hydrocarbon slurry reactor or gas phase agitated reactor.

Catalyst/product evaluation. Catalyst and product evaluation concentrates primarily on quantifying a smaller number of catalyst systems based on key performance indicators. This phase of research is concerned with quantitatively evaluating catalyst systems previously identified as promising.

Evaluation tests normally last longer than screening tests, and a smaller number of catalyst systems are evaluated as compared to the screening phase. The size is determined by the quantity of product required for evaluation and available quantities of synthesized catalyst. Usually, no attempt is made to mimic a commercial process because the catalyst and the product it produces are the main consideration.

Pilot plants are also constructed to evaluate the catalyst manufacturing process and provide scale-up data for the production of commercial catalysts.

Advantages in R&D

A growing number of polymer producers are recognizing the benefits of using pilot plants in their research and development programs. Unlike commercial plants, pilot plants are dedicated entirely to research, and the quality or throughput of products is not at stake. The best use of research and development resources can be made since the pilot plant is under the direct control of researchers.

The advantages of pilot plants versus commercial plants for process and product development, and technical service are many. Pilot plants, because of their small size, have fast start-ups and changeovers, and can recover quickly from operating problems. The quantities of off-spec or unsuccessful test products they produce are much lower than full-scale commercial units. Furthermore, the small size of the pilot plant minimizes the effects and risk of operation mishaps.

Scalable in size. There are many factors which determine the size and scope of a polymer pilot plant. An ideal pilot plant reactor should behave as much like a large-scale process reactor as possible, and yet duplicate or even extend the conditions studied in the laboratory.

Pilot plants used for kinetic studies and catalyst screening can be quite small because the quantity of catalyst available for testing is generally very low—a one- to four-liter batch system for bulk, solution, slurry, or solution processes is typical. Larger pilot plants may be required for handling minute quantities of highly active catalysts. In gas phase catalyst research the actual reactor and system is surprisingly large.

The minimum plant size for process development and technical services is determined by the degree of simulation required. The pilot plant needs to be large enough for a mass balance and to produce enough product to allow proper analyses. Also, the equipment needs to be large enough to have dependably controlled process parameters, such as mixing and temperature control. If downstream processing is critical, the pilot plant needs to be of sufficient size to feed the smallest commercially available equipment, such as centrifuges or extruders.

Product development pilot plants normally require the highest production rates and largest equipment. For market testing of a new product, or an enhanced existing product, enough product must be produced in a reasonable time so that several customers can use it as a test feed to their process. The most typical product rates are between 10 and 25 kg/hr. The largest units are able to make sufficient quantities of specialty products to partially finance their operation.

Design considerations. A pilot plant for a specialty polymer usually serves as an essential step between the laboratory chemistry and a new production process. The pilot plant is used to verify that the product developed in the laboratory can be produced successfully in larger, commercial equipment. For this reason, the design of the pilot plant reactor needs to be a small-scale model of the full-scale process equipment, even though the exact equipment that works best isn't known yet.

The pilot plant must be more versatile than any anticipated production equipment and be able to operate over a wider range of temperatures, pressures, viscosities and densities. During process development, higher temperatures or pressures may be necessary to provide desirable rates and yields. Often the primary influence on reactor design is a combination of high temperature plus high pressure or full vacuum. Temperature influences both material selection and equipment design—and combined with pressure or vacuum—makes the seals, valves and other components more difficult to design.

Viscosity is another important process variable for many polymerizations, especially those carried out in solution or bulk. Because of high molecular weight or low solvent content, the most economical process conditions tend to result in higher viscosities. In these cases, special impeller, mixer and reactor designs are necessary to handle the extreme viscosities.

Reactors are frequently designed to serve as general-purpose development tools and, as such, must be practical for both high- and low-viscosity polymers. Here, too, the unit's design must accommodate a wide speed range, different impellers, and other features.

The total chemical process of the polymerization has a considerable impact on the pilot plant equipment. Special metal alloys may be necessary to resist chemical attack as well as custom discharge valves leading from the reactor to a polymer pump when the polymer is processed directly into strands or pellets. In addition, modified batch equipment is sometimes needed for duplicating continuous processing conditions.

Source of process/product data

Polymer pilot plants, by nature, incorporate a wide range of instrumentation and control devices to measure production variables, supervise processes and collect product test data. In fact, control equipment represents a large percentage of the capital investment in a pilot plant. Instruments and sensors are used to monitor such critical process conditions as temperature, pressure, level and viscosity. Accuracy and reliability are of utmost importance in these systems, both in terms of conducting tests in the pilot plant reactor, and in gathering information that is useful for plant design and scaleup.

A unique characteristic of pilot plants is that the same sensors and controls are often used in both pilot- and commercial-scale equipment. Although instruments may be difficult

to install on small pilot reactors, they often can be applied directly to large-scale process equipment in a commercial plant.

To ensure greater accuracy and increased safety, modern pilot plants incorporate microprocessor-based control systems and intelligent transmitters, flow meters, pumps, thermocouples and other devices that communicate on a digital versus analog basis.

Digital control technology allows for unattended, stand-alone pilot plant operation and eliminates the need for log sheets and manual operation of the pilot reactor. In addition, it does away with concerns about operator bias and conflicts regarding data interpretation.

The integrity of digital process data is protected because its accuracy cannot be degraded by outside sources of interference. This is particularly critical when measuring low flow rates in slurries and solids, where digital metering equipment offers greater precision than standard analog-to-digital techniques.

Digital control systems also provide the pilot plant operator with complete control over feed rates, system operating pressure, reactor temperatures and sampling procedures. System software using an "open" architecture enables the operator to execute routines for data acquisition, logging, storage and historical trending, as well as set and change control parameters and alarm limits. These control routines frequently employ high-speed PID (proportional-integral-derivative) algorithms.

Conclusion

Because of the ever-increasing demands for better and more cost-effective polymerization processes and products, and tightening environmental regulations, innovative research is critical. Many producers of polymer products are finding that pilot plants are the answer to reducing development timelines, lowering operating and capital costs, improving product quality and minimizing waste streams.