

**FUTURE DEVELOPMENTS IN THE DESIGN AND USE OF
POLYOLEFIN PILOT PLANTS**

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Abstract

Rapid changes in the polyolefins industry and high-speed advances in polyolefin technology are changing research and development practices worldwide. As a result, the requirements for pilot plants used by researchers and engineers are changing, as well. This paper explores five areas of rapid change in the polyolefins industry: markets, industry, technology, methodologies, and information. These changes can be seen as main drivers for changes in polyolefins research and development and the future design and use of polyolefin pilot plants. After exploration of these five areas, a summary is made to envision the future pilot plants.

Polyolefin Pilot Plants

The very nature of polyolefin processes requires sophisticated pilot plants. The complexity of polyolefins in chemistry and transport, the difficulty in predicting the growth and behavior of particles through the process train, and uncertainties in predicting precise product properties dictate the use of pilot plants.

Pilot plants are justified on the basis of minimizing technical, economic, marketing, and production risks, and opening opportunities for more revenue and profitability. These uses and the need for material quantities for market testing can make polyolefin pilot plants in particular quite sizeable. Of course, these traditionally-sized pilot plants are costly to construct and operate. There is thus a drive to obtain more and better information at the laboratory scale: to make pilot plants smaller, more efficient, and of higher value, and even bypass piloting by more extensive use of modeling and simulation scale-up techniques.

The important changes now occurring in the marketplace and the industry, as well as in technology, will make concurrent changes in how we design and use pilot plants essential, and in fact inevitable.

Varied factors influence development

The question is how these developments will transform the polyolefin pilot plants of tomorrow – “pilot plants” being defined here as scaled-down continuous systems producing produce commercially relevant products and information. The answer is likely to depend heavily on five drivers: market drivers, industry drivers, polyolefin technology advances, new developments in research and development methodologies and improvements in simulation and modeling.

There is widespread agreement that market changes will have the strongest influence, as those will drive industry changes, which will drive the other three. At the same time, technology and R&D results will influence both the market and

industry, while themselves being accelerated by new R&D methods and simulation capabilities.

What remains to be seen is how all these drivers will influence polyolefin pilot plant design and use in the future. While it is impossible to accurately predict eventual outcomes, it is helpful to review what's in store as a result of each of the five major drivers. The future developments included in the following discussion serve as guideposts for advances, even if they are not an exact road map to the future.

Market demand for new materials prompt changes

Market demand will continue to grow for new materials with high performance capabilities in new or existing applications, for replacing more expensive or non-renewable natural and synthetic materials, and for replacing materials or processes that are associated with health and environmental risks. Environmental consciousness has increased market interest in recyclability and renewable feedstocks, as well. These and other market influences mean new materials research and development will remain a growth area for the foreseeable future, particularly for polyolefins.

The impact of technological advances such as the movement of olefin-based polymers and copolymers into both lower-end commodity polymers and higher-end engineering plastics applications is expected to significantly affect the polyolefin industry in the next few years. (Blum, 2001) Even now, the demand for new specialized materials has accelerated research and development of new materials and catalysts, and corporations are seeking rapid commercialization of such products to retain their competitive advantages. Metallocene-based polyethylene and polypropylene are seen competing with flexible PVC and elastomers. Metallocene-EPDM and ethylene copolymers are expected to displace other kinds of elastomers in some applications.

Polypropylene is especially viable as a source of new materials and applications. Metallocene-based polypropylene and its derivatives have moved it into the engineering resin markets and fibers, and it is expected to present a challenge, for example, to polyethylene terephthalate (PET). (Blum, 2001)

In higher-end markets, ethylene/carbon monoxide copolymer is finding use in high-service temperature plastic markets, and polycarbonate is being challenged by a metallocene-produced cyclic polyolefin, as indicated by Ticona GmbH of Frankfurt, Germany's commissioning of a plant in Oberhausen, Germany, to manufacture Topas – a cycloolefin copolymer. (Parkinson, 2000)

Polyolefin pilot plants will need to be large enough, flexible enough and sophisticated enough to meet increasing demands on researchers to develop new materials with new properties in quantities sufficient to demonstrate commercial production potential. Existing pilot plants may fill the bill where companies seek to produce the new polymers in existing commercial equipment. No doubt many new materials will require modifications to existing commercial and pilot

equipment, and when coupled with other the other factors discussed, a new pilot plant or partial replacement can become absolutely necessary for continued advancement.

Even simple market changes can require major pilot plant changes. The design pressures of bulk pilot reactors from ten years ago are sometimes insufficient to operate at conditions for the high concentrations of hydrogen required for high melt flow rate polypropylene. Thus new material demands and the desire to commercialize these new materials will drive new and modified pilot plants.

Biotechnology meets polyolefins

Perhaps more significant for future polyolefin pilot plant development is the market demand for environmentally acceptable plastics and renewable sources. That demand is spurring researchers to explore biodegradable polymers, renewable feedstocks from biomass for new polymers, duplication of commodity polymers and plastics recycling as feedstocks.

Such approaches, thought a pipe dream only a short while ago, are now reaching commercialization. Cargill, Inc. and Dow Chemical Co. have joined forces to commercially produce corn-derived polylactic acid (PLA) from food-grade sugar in a 140,000-metric-tons-per-year plant that is soon to start up. PLA is said to offer combinations of properties not available in hydrocarbon polymers. Future processes will endeavor to use inexpensive biomass feedstocks such as agricultural wastes and to compete with petroleum-based materials. (Parkinson, ed., 2000; Fairley, 2001)

Another indication that bioproducts' time has come is the production chain for polytrimethylene terephthalate (PTT). A joint venture between DuPont Chemical and Tate & Lyle Citric Acid will in the near future produce fermentation-derived 1,3-propanediol (bio-PDO), for a component of PTT. The union already has resulted in a bio-PDO pilot plant and is expected to start commercial-scale production by 2003. DuPont, meanwhile, has also commissioned a commercial-scale PTT plant and is working to encourage the market for PDO-based PTT, which it sells as Sorona. (Fairley, 2001) While these examples are not strictly polyolefins, the viability of biotechnology is indicated.

Such focus will require a completely new generation of pilot plant designs for polymers, involving enzymatic catalyst preparation and handling, novel feedstock processing and feeding, and entirely new reactor designs. In addition, pilot plants will be required specifically to study other new feedstocks derived from recycled plastics and their effects on traditional and new processes.

Industry drivers

Mergers and acquisitions, the resultant business and technology consolidations, increasing production and research costs, and patent and licensing constraints are driving many changes throughout the polyolefins industry (Moore, 1999), especially in research and development.

As corporations continue to merge and realign, a considerable pool of knowledge, equipment and funds for more advanced pilot plants is being created. Questions of risks and investments with today's very large plants are being weighed against the time and resources required for pilot plant work, with the likely result that the number, types, sizes and even design of future polyolefin pilot plants will be affected.

One inevitable result of mergers and consolidations is the duplication of existing research facilities and equipment, with an attendant shutdown of superfluous pilot plants and cancellation of some planned projects. For larger companies, mergers also mean additional resources for strong research and development programs to remain competitive in products and technology. As new and unfamiliar technology is acquired, producers are using multiple technologies worldwide and may find it advisable and possible to develop methods for meshing previously competing or non-interacting technologies, investigating catalyst-process, process-process and catalyst-catalyst permutations.

Smaller companies will have difficulty supporting pilot plant work while remaining profitable, and will need to license technology. Other firms will require pilot plants with special design features to prove patentable concepts. They also will be vigilant in seeking opportunities to skirt patented processes and catalysts to gain the same competitive advantages such patents afford the patent holders.

Rising energy and monomer costs are another industry issue that have been reducing margins. At the same time, R&D costs are rising. Research into cheaper monomer sources and use of new monomers will be stimulated, and energy-efficient processes will become more dominant, especially for commodities. In other cases, flexibility for high-value niche markets may be more important.

Taken together, these factors will result in pilot plants that will need to be highly flexible and designed with multiple technologies and catalysts in mind. Modularity in both function and design will be critical, both for multi-purpose plants and for the new generation of pilot plants that will be required to support new technologies, patents and licensing. These newer-generation plants will be specifically tailored to new technology requirements and will act in concert with or in addition to existing more flexible units.

To control costs and speed up commercialization, polyolefin pilot plants will preferentially use modular construction by in-house or, more typically, outside pilot plant experts. Barkey and Palluzi recommend modular construction as standard practice for all pilot and lab units. (Barkey, Palluzzi, 2000) Seeking outside pilot plant specialists in design and construction gives access to considerable experience with the problems and solutions unique to pilot plants and the small scale.

To further minimize the cost and time expenditures, pilot plants of the future will most certainly be more highly automated than their predecessors, and many will be smaller scale (less than 5 Kg/hr of product) with excellent commercial

relevance. These smaller continuous pilot plants will become workhorses for faster catalyst, process and product investigations at reasonable costs.

Another possible and interesting development is the idea of pilot plants owned and shared by a consortium of producers and technology companies, possibly operated by a third party. Technology confidentiality would be an issue in such a scenario.

One very large driver of future R&D is the likely economic leveling in production costs due to consolidation and diversification. This would make research and technology the new competitive benchmark for market and profitability. The demands of the polyolefin market coupled with industry changes mean that the economic opportunities, knowledge base, competitive pressure and financial resources exist to drive development of new polyolefin technologies, with interesting and revitalizing implications for the future.

Polyolefin technology developments drive business

Key to the market and industry changes is the advancement of polyolefin technology itself. Developments in single-site, Ziegler-Natta and other novel catalysts will continue and expand, with concurrent advances in processes to take full advantage of them. The result will be new processes and equipment – some of which may involve combinations of two or more existing process steps. (Caruana, 2000) With the new catalysts, means are available to utilize monomers and comonomers not possible before: incorporate polar monomers and comonomers, incorporate comonomers with functional groups, and reduce current comonomer requirements. In addition to the new materials such developments will offer, they can be expected to result in cost savings and opportunities to license technology.

A case in point is Samsung General Chemicals Co., Seoul, Korea, which is deciding whether to license the technology or sell the catalyst it has commercialized for producing high-performance polyethylenes. Production cost for Hybrid CG, as the catalyst is known, is about 25 percent that of metallocenes. The cost reduction is accomplished by reportedly reducing hexene requirements 30 percent. (Anonymous, 2000)

Recent process developments to maximize the use of new catalysts include condensed mode operations in gas-phase reactors, multiple reaction zones in a circulating fluidized bed, and operation at supercritical conditions. In the future, we may see use of new supercritical fluids, multi-zone reactors, incorporation of non-polymeric materials and bioprocesses, among other advances.

For many years polyolefin reactions have been carried out in bulk, slurry and gas phase using discrete CSTR, loop, fluid bed and stirred gas-phase reactors. When used in series, these reactors create separate phases that blend into the final polymer properties. Using a completely new reactor design concept – the Multi-Zone Circulating Reactor (Anonymous, March 2000) – Montell Polyolefins BV has intimately coupled the two phases by recirculating between two reaction zones in one reactor.

This is a revolutionary design, which Montell developed through the construction and use of a large dedicated pilot plant. The company plans a larger demonstration pilot unit to further commercialization. We can expect that the search for revolutionary capabilities will bring other novel designs that will require extensive pilot plant work for characterization and proof.

Other very unusual technologies for polyolefins are being developed at the laboratory stage. If they prove viable, they will require pilot plants with unique designs.

For example, work has been done successfully on polymerization of ethylene in a water emulsions phase (Soula, et al, 2000) and in supercritical carbon dioxide. (de Vries, et al, 2000) These potential future processes will require entirely new reactor and downstream process designs, which will in turn demand specialized pilot plants.

These developments and more will require new consideration of the equipment and scale-up for new pilot plants. The entirely new configurations and processes will require pilot plants of completely new and novel designs. The new materials from catalyst and process technologies will greatly expand the markets for polyolefins and improve profit margins, justifying future R&D expenditure on new technologies. The high demands for catalysts in the market will stimulate catalyst producers to move into high-speed materials research and even continuous pilot plants.

As the need for solutions to technical problems, accelerated research at lower cost, and technical competitiveness increases, we can look for new research and development methodologies to be incorporated into the polyolefin development process, with what are likely to be astonishing results.

Advances in R&D methodologies

Research and development methods and tools also are changing. Combinatorial methods, high-speed catalyst screening and product characterization, and development of more online analytical methods are prompting rapid investigations at the laboratory level. The increasing sophistication and capabilities of measurement and analytical instruments, especially for online use, will have a dramatic impact for polyolefin advances.

These developments will then have a direct impact future polyolefin pilot plants, as well. Some believe novel catalyst development time will be reduced from five years to as little as two years. (Anonymous, 2000), leaving pilot plant work a bottleneck to commercialization. Yet without pilot plant work, it is difficult to say with confidence that the selected combinations of catalyst systems and processes, from the myriad available to consolidated companies, are approaching the optimum revenue and profitability.

Many new automated analytical methods already are becoming available in the laboratory and for online monitoring and control functions in pilot and commercial

plants. They include new ways to characterize polymer products and to “look inside” reactors and process equipment.

Optical microscopy to measure single particle growth rate, high-resolution infrared spectroscopy to measure particle surface temperature and multipurpose kinetic calorimeter-reactors all are providing new insights into particle behavior during batch polymerizations. (Weickert, 2000) A continuous in-process imaging microscope is already available for online images of particle suspensions in a reactor. One in-process instrument based on laser technology can provide online information for the particle count, shape, and dimensions of small particles in slurry. This will most certainly be an important tool in future pilot plants. These in-process devices are sure to be incorporated in future bulk, slurry, and gas phase pilot and commercial reactors.

Electrostatic probes have been used to obtain useful information on agglomeration, sheeting, antistatic agents, and temperature control in fluidized bed reactors. (Servais, et al) X-ray Computer Assisted Tomography (CAT), 3-D multiple radioactive particle tracking and x-ray digital fluoroscopy are providing measurements of voidage, bubble growth and coalescence, solid circulation patterns and turbulence patterns. (Wright, et al, 2000)

These and other new means of “looking into the process” will certainly play a part in future polyolefin pilot plants, in conjunction with developments from information technology to incorporate them into online data interpretation and control.

Computing and information technology

From the previous sections of this paper, it is apparent that a lot of real-world data is now being generated. It has been suggested that we have been phasing out the “automobile era” and are now in the “information era,” with significant impacts on the process industries and chemical engineering. (Cobb, 2001) We could also be in a “new materials era.” Computing and information technology and their applications in the chemical industry have been rapidly developing, with the result that real-world data can be efficiently integrated and used. This is no less true for the polyolefins sector.

One highly active area in polyolefins information technology is the simulation and modeling of the polymerization process at all scales: molecular, microscale, mesoscale and macroscale. At the First European Conference on the Reaction Engineering of Polyolefins in July 2000, more than 25 papers were presented on simulation and modeling of the polyolefin polymerization process. Current modeling efforts are evident in molecular design of catalysts and the polymers they produce, product properties, kinetics and particle growth, particle heat and mass transfer, particle aggregates behavior (agglomeration, attrition, external heat and mass transfer), and reactors. A corresponding need for rapid investigation at the pilot plant level will affect control and data acquisition systems, flexibility and automation in pilot plants of the future.

There are some limitations to these models in application, including the numerous parameters that need to be determined. A rigorous model, even with simplification techniques such as Pareto diagrams, could require a year of pilot plant work to verify the model and parameters. Even then, it would be limited to the particular catalyst and application being modeled. Pilot plants will still be required to particularize them to specific processes and reactor schemes. (Cozewith, 2000)

One important area that models will impact is design. With the use of models and simulation, the scalability of difficult equipment and processes such as fluid bed and novel reactors will be greatly improved and the size of pilot plants needed for commercial relevance will be reduced. With modeling during development, considerable data exist to guide pilot plant modification or new design to more effectively use new catalysts and processes. Therefore, guided design with models will make pilot plants more effective tools that produce more useful data, faster.

A particular area of difficulty is scale-up of gas-phase fluid bed reactors. Very large pilot reactors are required for confidence, and it is believed that some modern technologies – for example, condensed mode operation – cannot be run effectively at the pilot scale.

Considerable progress is being made in modeling fluid bed reactors, however. (Yiagopoulos, et al, 2000) Future pilot fluid bed reactors could be designed for effective smaller-scale operation, with unique details that allow much more accurate scale-up. The pilot reactor design may actually be quite different from the commercial design, rather than simply a scaled-down commercial design, for most effective scalability.

Another very interesting possibility for reducing the amount of time needed for pilot plant work is development of dynamic models that can interpret online data without the long line-out period required to reach steady state in polymerizations. (Cozewith, 2000).

Models are being incorporated into process control schemes as “Model Predictive Control” (MPC) algorithms. When based on first principles these algorithms can be verified and tuned against pilot plant data rather than interrupting commercial operations. (Andersen, et al 2000; Schei, et al 2000) This is a new use for polyolefin pilot plants that will add to their value and justification in the future. Testing of and training for new advanced control systems and strategy can be done on simulators or on pilot plants for “real-world” experiences where the physical consequences are evident.

High levels of automation in control and data interpretation are definitely a characteristic of future polyolefin pilot plants. More automation is certain, with models incorporated in the control and data reduction schemes to produce more reliable data. Data integration with design and economic models will be standard. Signs of important things to come already are available, as for example the January announcement that BP and DSM are collaborating to develop high throughput experimentation tools, using robotics and other automation in almost every step.

With the addition of advanced controls, models, new measurement techniques, on-line visualization and analytical instruments, and their application to polymerization control and data interpretations, polyolefin pilot plant systems will look quite different in the future.

Looking ahead

In summary, the rapid pace of change in polyolefins will be the catalyst for significant changes in the pilot plants needed to support the market and industry. Based on the foregoing discussion, we can make some reasonable general predictions for the direction of future polyolefin pilot plants:

- ◆ The combined pressures of technological competition in the marketplace and eroded margins needing technological solutions will drive justifications for new pilot plants.
- ◆ Existing pilot plants based on current technologies will continue to be used extensively to commercialize the new products intended for production in the most popular existing commercial processes. If existing pilot plants are relatively new, they will be upgraded according to trends identified here. If they are older, with outmoded designs and systems, entirely new pilot plants can be justified, but may be delayed until the technology and consolidations picture clears.
- ◆ New pilot plants for existing processes will be based on expanded capabilities to encompass the extended process requirements of new products and take advantage of advances in instrumentation, modeling, and control systems.
- ◆ New online instruments for product properties, stream analyses, characterization of particles and particle systems, electrostatics, and direct information on particles inside the reactors will be extensively used for monitoring and control. New understandings based on this new data may lead to further breakthroughs in the technologies, and will certainly lead to better predictions of commercial performance.
- ◆ Large consolidated producers will have the considerable concentrated knowledge and means to build new multi-technology and multi-scope pilot plants with advanced features, and will do so to attain or preserve a leading technological position.
- ◆ New pilot plants merging catalysts, processes, and knowledge from previously existing but non-interacting technologies will result from industry mergers and technology consolidation.
- ◆ A new generation of pilot plants will be constructed for novel reactors and polymerization processes, such as multi-zone reactors, supercritical processes, and new reaction mediums. They will necessarily incorporate the advances in modeling, control, and instrumentation.
- ◆ Polymerization pilot plants incorporating biotechnology will become prevalent, incorporating new feedstocks, unique monomers and comonomers, enzymatic catalysts, and newly developed reactors and processes compatible

with the new technology. These will be required to develop an entirely new generation of biotech polymers for economic commercialization.

- ◆ Modular design, functionality, and construction will be critical to shorten lead-times, and for flexibility in the face of rapid changes.
- ◆ Small continuous pilot plants (less than 5 kg/hr) with excellent commercial relevance will be readily available and heavily used in development work.
- ◆ Models and simulations will be extensively used in designing scalable reactors and pilot plants, and will be directly incorporated into the control and data acquisition system, data reduction programs, and scale-up of the pilot plant results.
- ◆ Advanced control, including feedforward and model predictive control, will become prevalent in larger pilot plants to produce better data. The pilot plant programs will incorporate qualifying, testing, and training with advanced control schemes and models, provide model parameters, and calibrate model predictive control algorithms.
- ◆ Pilot plant control and data systems will be integrated online with laboratory, engineering, economic, and marketing information systems.

As this era of rapid changes – new materials, consolidations, technological competition, new tools – continues, the trends will play out in ways difficult to accurately gauge. Driven by market and industry trends, polyolefin pilot plants will keep step with such advances, providing the accurate, cost-effective data needed in the highly competitive modern world of polyolefins.

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