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Pilot Plants for Polyolefin Research and Development

by

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Abstract

This report discusses the choices of reactor engineering and sizing to be used in pilot plants for producing polyolefin resins in applications of research and development. By virtue of the collaboration between Oakwood Consulting, Inc. and Xytel Corporation, versatile and reliable continuous pilot plants are designed and constructed for the polyolefin industry. These are used to produce ethylene- and propylene-based resins, i.e., homopolymers, random copolymers, and impact ethylene-propylene (EP) copolymers, as well as a few others, such as terpolymers. The resin output capacity may be tailored to range from 2.0 to over 200 kg/hr. The small-scale continuous units may be models of world-scale commercial facilities--models that may optionally include catalyst mixing, prepolymerization, and metering into a train of multiple polymerization reactors. Design flexibility also permits one to create a unique configuration for a conceptual process, if desired.

The primary focus of this presentation is on suspension types of polymerization reaction environments. Some common examples are the following: polyethylene (PE) made in diluent hydrocarbon slurry, polypropylene (PP) made in bulk (liquid-pool) monomer, and both polymers made in powder-gas phase reactors of various designs. Depending on the application to be emphasized, the size is chosen: catalyst evaluation tending to be smaller units and market evaluation lots tending to be very large units.

The scale-down and scale-up guidelines are discussed and tabulated quantitatively for some common examples of reaction environments, using scale factors ranging from 5 to over 4000. Principles used in scaling include geometric factors, dimensional factors, and dimensionless factors. Issues and guidelines for heat and mass transfer are included in these tables. Some examples of pilot plant operating data are included to demonstrate the application of these units to measurements of Melt Index response to the reactor concentration of hydrogen and the resin density response to the concentration of comonomer used. The pilot data are extrapolated to include the application to full-scale operation.

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I. Introduction -- Scope and Concept

The polyolefin industry has reached a stage of sophistication in which profitability requires a stream of new products that are differentiated from the commodity resins of the past. The enhanced physical properties of new resins should command a higher profit margin. In order to develop these new resins, industry must rely heavily on effective research tools: experiments, models and pilot plants (Schlegel, 1972). In the words of the late Dr. Leo Baekeland, inventor of “Bakelite” phenol/formaldehyde resin, concerning the value of pilot plants: “Commit your blunders on a small scale and make your profits on a large scale” (Griskey, 1979)

In most cases, the starting point for R & D consists of experimental work to obtain reaction kinetic data on a laboratory scale, in which both the apparatus and the reagent quantities are very small. Such work may be done in a test tube or a beaker, which may not resemble a commercial reactor. Another approach is to make use of process models. One such model is a mathematical model -- an empirical or theoretical equation-based description of all key process features and reaction kinetics. The challenge with such a description rests in the qualifiers “all key”. It is a rare situation in which “all key” features are ever known, especially in the early stages of process development. Xytel and Oakwood use mathematical models as an adjunct to the design and construction of pilot plants (Brockmeier and Rogan, 1985). Another model could be an exact physical replica of a large scale process. However, since the changes in the relationships of process parameters change differently for different scales, it is impossible to have such an identical replica. Some dimensionless parameters may be satisfied while others are not.

II. Pilot Plant Reactors for Polyolefins R & D

This presentation emphasizes another research tool -- the pilot plant -- a small-scale production facility that **resembles** a full-scale prototype. A pilot plant incorporates similarity relationships that are judged to be the key engineering challenges in the process -- such as: heat and mass transfer, polymerization reaction kinetics, reactor residence time, particle formation and behavior, flow characteristics, distribution of residence times, and process dynamics (Calderone, 1994).

The body of this presentation will focus on polyolefins made in various suspension-type reaction environments using continuous pilot plants. One very well-known class of process is based on slurry suspension in a liquid pool of monomer, or a mixture of monomers that may be dissolved in an inert solvent. Another popular process is based on powder fluidization in recirculating monomer vapor phase.

There are a number of reasons why industry experts rely on pilot plants and models. Those who allocate tens of millions of dollars to a commercial project want reliable design information to increase their scale-up confidence. Depending on many issues, a typical scale-up factor may fall in the range of 100 to 1000. For an existing business, a pilot plant is valuable to provide technical service regarding plant operating concerns. New products are developed from the tests run in a pilot plant. Significant quantities of resins are made for market testing and evaluation. Operating campaigns provide vital data for safety and environmental studies. Modifications to the small-scale equipment provide the know-how to do new process development. Catalyst variations are readily tested to evaluate their merit.

II. Discussion, Design of a Facility

Some popular types and sizes of polyolefin pilot plants and their applications are summarized in Table I. Depending on the size selected, some applications are favored over others. These facilities are versatile in being able to (optionally) use any of the common high activity catalysts: Ziegler-Natta, Single-Site, and Metallocene types. Given the focus on suspension (rather than solution) polymerization, each of these catalysts will be carried on a suitable support and fed as heterogeneous fine particles in a carrier. The first row of Table I lists the smallest units, for laboratory-scale batchwise operation, having a production capacity in the range of 0.5 to 2.0 kg per batch of polyolefin homopolymers. Consisting of a single reactor, the operating mode can be a resin slurry in either a hydrocarbon carrier or in liquid monomer. These units are most suited for catalyst screening, for determining catalyst kinetic parameters, and for qualitative evaluation of resin physical properties.

Table I. Types and Sizes of Pilot Polyolefin Reactors

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The second row of Table I shows a continuous pilot facility that has somewhat greater production capacity, ranging from 2 to 5 kg/hr (Brockmeier, Arzoumanidis, Townsend and Lawlor, 1998). With two or more reactors included, the various types of possible resin products include more variety: homo-, co-, and ter-polymers. The middle column lists the various reactor choices that are typical. In this small capacity range, the continuous stirred tank reactor (CSTR) is often selected. There are scale-down challenges in trying to use liquid-propylene loop reactors or gas-fluidized bed reactors in this range. Axial-flow elbow pumps are not readily available in suitable small sizes. It is not cost-efficient to select a larger size gas blower and then modify the configuration to use a very small fluidized bed, and large scale-up factors for fluidized beds have not been reported to be reliable. A continuous unit in this size range is ideally suited to do catalyst evaluation and to study the melt index response to hydrogen. Such a unit size is large enough to make sufficient resin for physical property evaluation, to study kinetics, and to provide operating data for scale-up.

One example of a small continuous pilot plant is shown in the drawing of Figure 1, which is a unit designed to make propylene-based products in a series of two bulk reactors, followed by a gas-phase reactor. It has a product slate of homopolymer, random copolymer, and impact ethylene-propylene copolymer, up to 5 kg/hr output.

Figure 1. 5 Kg/hr Continuous Polymerization Unit for Polypropylene

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The last row in Table I extends the range of production capacities from 10 to over 200 kg/hr. There are pros and cons to the choice of such a semiworks facility. Obviously, the capital and operating costs are much greater than those for the smaller sizes shown in the previous table. The use for catalyst evaluation and for reaction kinetics generally will be more costly and less efficient. On the pro side are several features: At this scale, loop elbow pumps and centrifugal gas blowers are in a more typical operating range. This allows R&D to operate a pilot facility that is more nearly a miniature model of a full-scale prototype -- a feature that generally is perceived to reduce the technical risks involved in scale-up. Of course, this size range usually provides greater confidence for a scale-up to commercial-sized polyolefin plants, by virtue of the reduced scale-up factor.

These latter factors point to two well-recognized advantages of the large-scale pilot plant: the ability to produce test quantities of resin that can be run in a customer's full-scale extruders and other processing equipment; and the ability to do a better job of plant technical service to solve operating problems that develop. Catalyst preparation may be done batchwise, although it is usually preferable to do a continuous prepolymerization for pilot facilities larger than 20 kg/hr capacity (Brockmeier, Arzoumanidis, Karayannis, and Stein, 1996). In either case, mixing and metering of each of the catalyst system components is necessary. For Z-N, the system includes three components in three streams: The catalyst mix, the alkyl cocatalyst, and the donor that governs the stereoselectivity for isotactic PP. Figure 2 shows a very large continuous polyolefin pilot unit used for making either PP or PE resins.

Figure 2. Very Large Continuous Multipurpose Polyolefin Facility

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III. Scaling Principles for Pilot Reactors

Experience has taught design engineers to follow somewhat different scaling guidelines (Figure 3) for each unique type of pilot plant, usually associated with the choice of reaction environment. These

Figure 3. Key Scaling Parameter Guidelines for Continuous Pilot Plants
Equivalent Residence Times

Pipe Loop	Volume L/D Partic

guidelines change somewhat with the choice: Pipe loops, stirred tanks, or gas phase/powder beds. The gas phase beds differ very much from each other, depending on the powder agitation intensity-- whether very mild mechanical stirring or, at the other extreme, an intensely fluidized bed.

If the design scaling has been done correctly, polymerization on the suspended heterogeneous catalyst particle does not depend on the macro-scale reactor, but only on the micro-scale environment surrounding each growing resin particle. Heat transfer and monomer transfer must be controlled on the micro-scale in order to produce free-flowing resin particles, no matter what scale of reactor size is employed. This principle also allows the designer to model (approximate) a large pipe loop reactor on a very small scale by using a CSTR. With caution, the same can be said for approximating a large fluidized bed.

Pipeline Reactor Scaling:

Table II lists the key scaling parameters for pipeline reactors in which polymerization occurs on particles suspended in liquid propylene circulating around the loop in highly turbulent flow (Zacca and Ray, 1993; Samson, Weickert, Heerze, and Westerterp, 1998). The Reynolds number of 720,000 (and high linear velocities) are more than sufficient to overcome the tendency of 1000 micrometer PP particles to settle, and to achieve an overall heat transfer coefficient in the range of $2.0 \times 10^6 - 2.9 \times 10^6$ Joule/hr-sq m-°C. High velocity cooling water in the jackets removes the heat of reaction to maintain a temperature of 160 - 170 F. The table shows parameters for five different scales: 5, 25, 75, 250, and 7500 kg/hr production capacities. In each case, the reactors are operated with a mean residence time of 1.2-1.5 hr at a slurry concentration of 50 wt percent resin solids.

Table II
PIPELOOP REACTORS, DESIGN SCALING FACTORS

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The first column on the right lists the key parameters for a world-scale double pipelooop capable of making 7500 kg/hr. The geometric parameters are listed near the top of the column: the surface-to-volume per m (S/V); the length-to-diameter ratio (L/D); the length of the loop in meters, L; the pipe I.D. in meters, D; and reactor volume in cubic meters. The designer is immediately met with the challenge of how to deal with S/V as the scale is changed by a hundred-fold to fabricate a small unit.

The dimensionless Reynolds number (Re) is listed to indicate the high turbulence -- in this regime, heat transfer is not a strong function of Re.

The dimensional parameters are more restrictive to the designer. The slurry viscosity, which is a function of slurry concentration and other factors, strongly affects the circulation and settling velocities. The circulation pump must provide sufficient head and flow to achieve the desired fluid velocities. The fluid properties and geometry determine the values of the five thermal parameters at the bottom of the column. It is no surprise that the change in S/V is reflected in the thermal parameters that are based on heat transfer area: kg/hr-sq m and Joule/hr-sq m.

Move to the second column from the right, the one that lists the designer's choices for a 30-fold scaledown from the commercial-scale plant of 7500 kg/hr. The reactor volume is also reduced by 30-fold so as to maintain constant residence time at the same operating conditions and catalyst productivity. The S/V rises by a factor of three, to a value of 20. Because of this increase, the cooling load per unit area on the jackets is reduced by a factor of three. The fluid velocity is increased by the same factor to maintain the same Re and heat transfer coefficient.

Next, note the parameters in the third column from the right in Table II, which lists the designer's choices for a 100-fold scaledown from the larger full-scale plant of 7500 kg/hr. The reactor volume is also reduced by 100-fold so as to maintain the residence time at the same operating conditions and catalyst productivity. The S/V rises by a factor of four, to a value of 26. Because of this increase, the cooling load per unit area on the jackets is reduced by a factor of four. The fluid velocity is increased by the same factor to maintain the same Re and heat transfer coefficient.

Move to the fourth column from the right, which lists the design parameters for a 300-fold scaledown from the world-scale reactor size. The pipe loop diameter chosen for this is 0.1 m. At this small diameter, there is a challenge as to the availability of a 0.1-meter axial-flow elbow pump. A common choice is to specify a 0.15-m elbow pump, including 2.4 m of 0.15-m pipe, with the 11-meter balance of the reactor length being 0.1-meter diameter. This production capacity is just about the limit of scale-down for a true pipe loop reaction system. For this small diameter pipe, a slurry velocity of 3.7 m/sec is sufficient to maintain a good suspension. Furthermore, these small axial-flow pumps are being stretched to the limit of their operating fluid head. For a pilot plant of smaller capacity, the

designer's choice probably would be a CSTR, or a pair of CSTRs in series to approximate the behavior of the pair of pipeloops.

The parameters listed in the second column from the left in this table show what a 1500-fold scaledown of a pipelooop reactor would be: It is a little like taking a 0.37-m length out of a 0.15-meter pipe and operating it as a CSTR. The S/V for this CSTR is about the same as for a 0.15-meter pipe, 20 to 26 per meter. However, the miniature CSTR must rely on external cooling, such as a condenser, in order to achieve the 290 – 700 kg/hr-cu m productivity comparable to a pipelooop.

Gas-Fluidized Bed Reactor Scaling:

Another popular choice for polyolefin production is fluidized powder gas-phase reactors (Samson, van Middelkoop, Weickert, and Westertep, 1999). These are sometimes single reactors for polyethylene and polypropylene homopolymers, or multiple reactors placed in a series train, especially for polypropylene copolymer production. Table III (next page) shows the design parameters for a single propylene homopolymer reactor of 22000 kg/hr capacity. Just as in the case of pipelooop geometry, there are the following parameters referring to: geometry, dimensionless, and dimensional parameters such as residence time. Operation of the powder bed, which is agitated by gas flow, is best in the L/D geometry range of about five to seven. With bed cooling achieved primarily by an external heat exchanger/partial condenser, the reactor S/V is not a meaningful parameter. Neither are the thermal parameters calculated on the basis of per square meter -- the heat transfer area is in the external exchanger, not the bed. This kind of gas-phase reactor (without supercondensing) has only about half the thermal capability of the pipelooop reactor.

This table also lists fluid bed reactors for 4800 and for 150 kg/hr scales -- parameters in the other columns show the dimensions for fluidized bed reactors that are scaled down by factors of 4.6 and 147. Note that the smallest scale reactor in Table III is a substitution of a 1 - 2 kg/hr CSTR to model the behavior of a vertical bed fluidized by gas.

Table III
GAS FLUIDIZED REACTORS, DESIGN SCALING FACTORS
(Non-supercondensing)

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Stirred Tank Reactor Scaling:

This provides a segue into those commercial polypropylene processes that are still in operation using CSTRs. The first column on the right in Table IV lists the design parameters that pertain to a 94.6 cu meter stirred tank that can produce 15,000 kg/hr of PP operating at 45 to 50 wt percent slurry concentration at roughly the same conditions of temperature and pressure used in the pipeloo reactors. The cooling capability of such a jacketed reactor is only a small fraction of the value achieved in a jacketed pipeloo, so external cooling is needed -- often an overhead reflux condenser is used.

The parameters listed in column two of this table show what a 3000-fold scaledown of such a CSTR would be: It is a CSTR 0.18 meters in diameter by 0.36 meters high. The S/V for this CSTR is about the same as for a pipeloo, 20 - 26 per meter. However, the miniature CSTR also must rely on external cooling, such as a condenser, in order to achieve an 290 - 320 kg/hr-cu m productivity comparable to a pipeloo.

Table IV
STIRRED TANK REACTORS, DESIGN SCALING FACTORS

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The operation of such miniature pilot plants having production capacities in the range of from 2 to 5 kg/hr poses several challenges in addition to the necessity of approximating the geometry of one reactor with that of another. Accurate metering of the very small flowrates of various streams is another challenge. This is made especially difficult by virtue of the desire to make resin particles that fall in the size range as that of the commercial product resin, ie., 800 to 1000 micrometer diameter. To relax this requirement would defeat the goal of having pilot plant operating results that are commercially relevant. Recall earlier comments regarding the micro-scale of reaction. Use of valves and lines sized to continuously throttle the typically low flowrates usually fails on such a small scale -- flow should be intermittent.

Agitated Powder Bed Reactor Scaling:

Another choice for polyolefin production is the agitated powder gas-phase reactor. Table V shows the design parameters for a single propylene homopolymer reactor of 4000 kg/hr capacity. The same design parameters are used, referring to: geometry, dimensionless, and dimensional parameters such as residence time. Operation of the powder bed, which is agitated both by gas flow and an impeller, is best in the L/D geometry range of about five to seven. With bed cooling achieved primarily by an external heat exchanger/partial condenser, the reactor S/V is not a meaningful parameter.

Table V
AGITATED GAS-PHASE HOMO-REACTORS, DESIGN SCALE FACTORS

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Table V lists mechanically fluidized reactors sized for production capacities of 4000, 1600, and 37.5 kg/hr. The dimensions are shown for agitated gas-phase reactors that are scaled down by factors of 2.5, 107, and a factor ranging from 2000 to 4000. Note that the smallest scale reactor is a substitution of a CSTR to model the behavior of a vertical bed fluidized by gas and agitation.

IV. Operating Results

The operation of a polyolefin pilot plant provides the opportunity to obtain experimental results that should answer most of the following important questions:

Will the catalyst work in this larger scale?

Can the resin grade (MI or MFR) be made with suitable physical properties in this reactor?

Will the chosen control strategy provide stable operation of the unit?

If there are recycle streams, what happens as entities accumulate in the process?

Are the results relevant for scale-up to commercial operation?

The following figures and tables show and compare operating data that have been obtained both from pilot plants and from full-scale plants. The sources were two producers who have Xytel pilot plants and supplied us with both pilot and commercial data.

A 50 kg/hr hexane slurry High Density Polyethylene (HDPE) pilot plant built by Xytel is diagrammed in Figure 4. A pair of CSTRs in parallel feed resin slurry to a degasser to concentrate the solids, which are then separated in a centrifuge and dried. The hexane is recycled. The operating conditions for this unit are: Temperature= 105° C, pressure= 4.5 Mpa (10^6 newton/sq m) using a new supported Ziegler-Natta (ZN) catalyst having a weight-ratio composition of $TiCl_4$:TEA:MgCl₂ approximately equal to 1:6:40 (TEA is triethyl aluminum alkyl). Answers are obtained for each of the five major questions above and summarized in Table VI. The first two rows in the table pertain to

Figure 4. Hexane Slurry Process for HDPE

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catalyst activity. Based on the catalyst productivity measured in the pilot plant, a prediction is made for productivity at the operating conditions of the commercial plant, which is estimated to be 300-fold larger in capacity. For the commercial plant, note the close agreement between predicted and actual productivity in kg-PE/g-catalyst, for both the first reactor and the second reactor. Productivity is the inverse concentration of catalyst residue determined both by material balance and by sample incineration (such as by r.f. heating). One can conclude that the catalyst will work and also that contaminants do not accumulate in the recycle so as to poison the reaction. With the use of the new catalyst, residues are lower and off-specification production is also reduced.

Table VI. Results for Applying New Catalyst

Hydrogen is the chain transfer agent used to limit the degree of polymerization to achieve the desired Melt Index (MI) of the resin grade being made. Table VI shows for each reactor the MI response from operation at a given molar ratio of H₂-to-C₂H₄ monomer -- the actual commercial result is about 6% higher than the predicted value for the first reactor. For the second reactor, there is a greater discrepancy--not surprising at the very low MI values. The resin MI can be reproduced in the full-scale plant and the control strategy appears satisfactory. It is also reasonable to conclude that the response to hydrogen provides satisfactory sensitivity to control the MI.

A key property for the copolymer from the second reactor is the resin density, which is controlled by the targeted level of comonomer used. The bottom row in Table VI shows the very close correspondence between predicted and actual comonomer response of density -- within about 1 percent. The graph in Figure 5 shows several experimental operating points for this response of density, including tests both with a previous catalyst and with the new catalyst.

Figure 5. Comonomer Response

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Another example follows of the use of a pilot plant to answer key questions (see above) about the operating behavior to be expected from a commercial-scale plant. In this instance, a linear low-density polyethylene (LLDPE) is made at typical conditions by Equistar in a well-known gas-fluidized bed reactor process, using ethylene and a small fraction (1 to 5%) of a suitable alpha-olefin comonomer, such as butene-1. The operation is at $T = 90^{\circ} \text{C}$, $P = 2.5 \text{ Mpa}$, using a supported ZN TiCl_4 catalyst that is similar to the one cited above for HDPE. The commercial reactor is roughly 400 times larger in working volume than the pilot plant reactor. However, the commercial reactor is operated with a relatively higher production rate, such that its mean residence time is 1.5 hours, rather than 2.4 hours used in the pilot unit, as summarized in Table VII below. Activity is defined as productivity divided by the mean residence time.

Table VII. Linear Low-Density Polyethylene Made in a Gas-Fluidized Bed

The operation of the supported Ziegler-Natta catalyst appears to be typical, given the lower residence time resulting from higher throughput: these catalysts typically decay in activity with increasing residence time, as the tabulated operating data show. This reduced activity is probably not a result of poisons or inerts accumulating in the system with recycle, but is more likely due to loss of active sites. Commercial productivities in the range of 5000 or more are quite acceptable in the industry. Though not obvious from what is included in Table VII, the process control strategies are acceptable on both scales of operation because the units achieved steady running conditions.

One of the very important concerns centers on whether the same resin grades can be made at predictable conditions both in a pilot plant and in the commercial-scale plant. The last row in Table VII shows that the resin MI made at either scale of operation is closely correlated to the target molar ratio of H_2 -to- C_2H_4 monomer: a target of 0.15 to 0.16 will yield an MI of 1. A target of 0.22 +/- .01 will yield an MI of 2. All four resin samples have the same density: 0.918 g/cc. Other physical properties of the resin, while very important, are outside the scope of this report.

Both of the examples cited above demonstrate one of the most important advantages of a well-designed and smoothly operating pilot plant: The continuous mode of operation yields operating data that are *relevant* for reliable scale-up from the pilot scale to world-scale commercial plants. Small laboratory units, which are typically batch-wise in operation, simply cannot accurately measure results at a constant set of conditions, such as MI response to hydrogen, because concentrations change during the course of a polymerization. Lab-scale operation also cannot make volumes of resin needed for a

commercial evaluation, nor does it work well to analyze the accumulation of contaminants with recycle. The results from a reliable pilot plant generate *confidence* in the minds of the decision-makers.

V. Conclusions

We can conclude that there is a organized body of mathematical relationships that is a valuable guide to the design and engineering of small-scale pilot plants. These scaling principles are specific to the type of polymerization process that is being considered. Polyolefin pilot plants that have been constructed according to these principles by the Xytel Corporation are running successfully lined-out to obtain operating data that are relevant to the operation of full-scale commercial polyolefin plants: whether in a gas-phase or in a liquid-phase (slurry) reaction environment.

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