Pilot testing services for a wide range of unit operations

The power of testing

The state-of-the-art test centers of Sulzer Chemtech's business unit Process Technology offer customers pilot testing services for a wide range of single units of operation and combinations thereof. A comprehensive number of separation and polymer reaction units, highly skilled test center engineers, and analytical equipment are all available for tests of systems on a pilot scale. This testing can validate the assumptions made in process design and supports customers with their development projects.

Process simulation has progressed immensely. Comprehensive electronic databases of physical properties have become available for pure components and for mixtures. All of these data can be accessed with little effort from personal computers. Chemical and process engineers have been able to greatly improve their efficiency and productivity thanks to these tools, user-friendly graphical interfaces, efficient codes, and broad correlation libraries. Today, alternative designs and hybrid process solutions that combine units of operation and parametric-sensitivity analyses can be obtained rapidly—which enables engineers to offer increasingly complex optimization solutions.

In 1966, Thomas Sherwood, a distinguished professor of chemical engineering at MIT, emphasized that any engineering design process involved making assumptions[1]. The options he proposed for obtaining missing data were: asking the company's laboratory, building a pilot plant, or purchasing the know-how. Several decades later, the tools have evolved but this description remains very accurate.

1 Thin-film evaporator.

Diverse needs for pilot testing

From an economical point of view, pilot testing becomes necessary when it is more expensive to cover the uncertainty of the assumptions with reserve than to run a pilot test. However, there are also cases where the range of the uncertainty cannot be quantified.

Rectification is a separation technology that has been intensively studied. Nowadays, many chemical separations can be properly simulated without the need for any testing. After engineers have identified the frame of the uncertainty, some design margins can be built in—typically, by increasing the reflux ratio, adding more trays, or lengthening the packed beds. The concentration of aqueous hydrogen peroxide by evaporation and rectification is a good example of a system that can be quite well pre-

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dicted. Purifications might be challenging to simulate when the feed contains many different chemicals.

During the sizing of rectification columns for products from coal tar, it is not unusual to find over 100 peaks on the chromatogram of the feed. Some exotic chemicals are not even found in common databases. In such cases, it might be necessary to run a validation test for the simulation of the rectification. This is especially true when rectification is required to lower the concentration of specific components below a threshold value because of their toxicity and where there is limited confidence in the simulation results.

The process of evaporation as a unit operation is reasonably well understood. The challenges related to the modeling of the vapor-liquid equilibria are similar to those in rectification. Film evaporation is often used for processes under vacuum and for handling feed with higher viscosity. Piloting might be needed to optimize and verify the concentration of residual low-boiling impurities in a high-boiling product or the losses of low-boiling product when evaporated out of a higher-boiling purge [1].

Additionally, pilot tests allow the validation of the calculated overall heat transfer coefficient.

Pilot testing is also necessary for separation by fractional crystallization. This staged separation technique relies upon solid-liquid phase transition and on the virtue of selectivity found in solid-liquid equilibrium. It allows multicomponent mixtures to be split into narrow fractions, ultimately leading to top purities of selected components. Although the solid-liquid phase equilibria are known for many binary and even ternary systems, it is still not possible to precisely predict the separation without running pilot tests.

One of the challenges is the effect of the crystal habitus (shape), which can strongly influence the ability of the crystallized mass to hold back part of the mother liquor with the impurities[2]. The pilot test results allow for a reliable design of an industrial unit. The purification of acrylic acid to the "glacial" grade is a typical example, as shown in figure [2].

A different situation with a similar consequence is found in the scale-up of liquid-liquid extraction columns. This technology provides many advantages, such as low energy consumption and the possibility of concentrating and purifying chemicals that are diluted in a bulk liquid. Agitated columns are preferred for many applications because they allow for controlled energy input in the agitated sections. In most of the cases, however, pilot tests should be performed to verify that the system is behaving as the liquid-liquid equilibrium data have predicted.

Some mixtures contain salts or impurities that may affect the interfacial tension and thereby strongly influence the specific interfacial surface and phase separation. Through pilot tests, agitated columns [3] have been successfully scaled up to 3.1 meters in diameter. For larger volume flows, packed columns are often preferred because of their higher throughput. The diameter of a liquid-liquid extraction column can be scaled up with good reliability as long as the axial backmixing is properly considered. A test in a pilot column is often necessary in order to verify this phenomenon[3].

[3] Agitated liquid-liquid extraction column in pilot scale.

[2]a and [2]b Acrylic acid crystallization unit and pilot plant for crystallization.

Membrane separation processes, such as pervaporation and membrane filtration (reverse osmosis, nanofiltration in aqueous and solvent mixtures), are receiving increased attention in the industry because of their ability to cross azeotropes and because of their energy efficiency[4)].

Membranes have complex structures, and innovation is very dynamic in this promising field. The fundamentals of membrane design have been addressed[4)], but the specific throughput and selectivity of a membrane for a given mixture is seldom precisely predictable. Since the necessary surface area of a membrane has a strong impact on the investment of the plant, pilot testing is generally advisable. The testing with membranes allows the measurement of selectivity and throughput. It is also possible to test the effect of impurities on the membrane's performance through long-term stability tests.

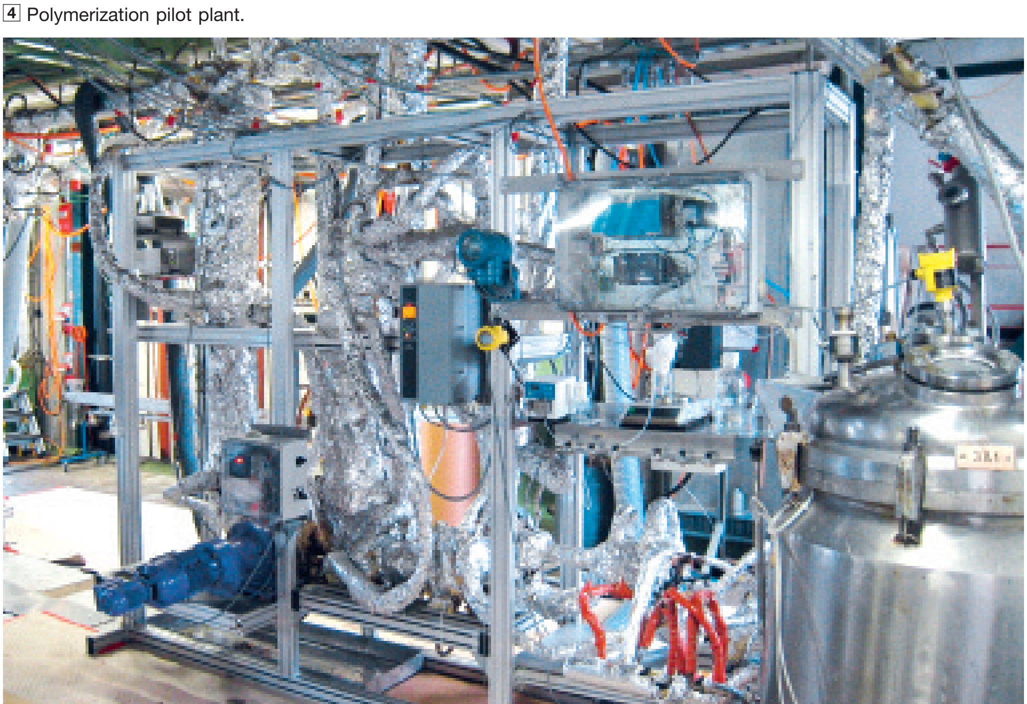
The challenges in processing might go beyond concentration and purification issues. Often, products prone to coloration—like triethanolamine or unsaturated fatty acids—are processed. Different solutions can be applied to avoid or minimize color formation depending on the mechanisms affecting this.

Examples are: oxidization due to air (leakage) or other oxidants present in the feed, thermal cracking at the hot walls of an evaporator, or thermal cracking in the bulk if products are exposed to longer residence time. When experience is lacking, a pilot test can be run in a falling-film evaporator, a thin-film evaporator, or a rectification column to verify the color of the end products. Sometimes, for instance in the cosmetic industry, it is necessary to remove unpleasant odors. Odors might, on the contrary, need to be concentrated, as in the beverage industry. The molecules responsible for the odor usually cannot be simulated as chemicals. Unless experience already exists for the application, it is necessary to run pilot tests to reliably predict the outcome.

Pilot testing is commonly needed in polymer technology for the design of specific production processes and for the scale-up of equipment 4. In these polymerization processes, the viscosity changes by several orders of magnitude. It can increase by factors beyond 10^6.

The prediction of flow behavior and phase equilibria becomes very challenging. It is necessary to test on pilot scale, in particular, for new polymers and elastomers, prior to designing an industrial unit.

4 Polymerization pilot plant.



The requirements for pilot test facilities

Large companies are usually well equipped with their own testing facilities. Standard equipment generally includes stirred tank reactors, rectification columns, evaporators, and liquid-liquid extraction columns. Depending on the market segment, specialized technologies, such as membrane separation rigs, are implemented. The challenges of having in-house facilities are mainly in the control of costs and the maintenance of the expertise.

It is expensive to own all possible testing rigs. Rental units are sometimes a good alternative in these cases. Nevertheless, safety measures, personal protection, and environmental regulations add to the costs required to finance pilot plants. In addition to the pilot rigs themselves, analytical devices, a chemical storage system, and a ventilation system are necessary. Often, the pilot plant installations have to be explosion-proofed, which requires the use of even more expensive equipment.

Special attention has to be paid to proper installation regarding leak-tightness and insulation. Because pilot and mini-plant units exhibit low liquid hold-up volumes and relatively large surface areas, leakage and heat losses have a much stronger impact on the test results than in industrial-size units. In order to minimize these effects, advanced heat tracing and expensive sealing systems have to be employed.

The companies running polymerization processes face similar issues. The ownership of a pilot plant is often limited to a stirred tank. The implementation and operation of a loop reactor and a finishing section represent expensive investments for occasional process development.

Another prohibitive aspect of in-house testing is the level of competence of the operating personal. Pilot testing should verify the assumptions and increase the confidence in the process design. Ultimately, the pilot test will set the basis for the design of a large-scale unit. An unrecognized mistake in the test can lead to disastrous design errors and financial losses. The staff running the test must, therefore, be well trained. Long years of experience and practice have great value.

The correct operation of the control loops illustrates this issue. In a large plant, it usually takes a week or more to properly tune the control loops. In a pilot test, the time and the amount of feed available are limited. Therefore, the tuning must be completed within a few hours.

Innovation leads to new solutions, hybrid processes, and, more generally, process intensification. The combination of technologies, such as distillation and pervaporation, liquid-liquid extraction and stripping, or evaporation and crystallization is quite complex to perform.

The knowledge and experience necessary to operate such combinations at the pilot scale are seldom available in chemical plants. A further issue is the necessity of owning and maintaining all the pilot plants for the diverse technologies. Finally, it is often advantageous to have different pilot plant scales for a single technology.

Customer testing facilities at Sulzer Chemtech

Sulzer Chemtech acquired the company Kühni in 2009. The existing process solution competencies were combined into a single business unit named Process Technology. The unit offers process design, engineering services, process equipment, modular plants, and skids for a wide range of process solutions. It also covers pilot testing for all available unit operations, and it provides the opportunity to combine them.

The following pilot units and analytical equipment are available at various locations 5:

- Distillation
  - 7 columns Ø 30 mm up to 500 mm
  - batch or continuous
  - bubble cap trays, slit trays, structured packing, and spinning band
- Evaporators
  - 3 falling-film evaporators
  - 3 thin-film evaporators 1
  - 2 short-path evaporators
- Liquid-liquid extraction
  - 4 columns Ø 32 mm – 150 mm 3
  - 3 mixer-settlers Ø 40 mm, 3 mixer-settlers Ø 100 mm
- Membrane technology
  - benchtop systems
  - pilot units
  - pressure-driven membrane pilot-scale unit
- Crystallization
  - 3 falling-film crystallizers 2
  - 2 static crystallizers
  - 1 suspension crystallizer
- Polymerization technology
  - static-mixer reactors for the polymerization of various monomers, e.g., styrene and lactides (lactic acid diester) 4
  - degassing rig for devolatilization
  - expandable polystyrene (EPS) pilot line
  - various extruders and static mixers for additive mixing tests
- Analytical capabilities
  - GC-FID, GC-HWD, headspace GC
  - HPLC
  - UV-VIS spectrometry
  - titration (acid-base, Karl-Fischer volumetric and coulometric)
  - solid or moisture content by thermobalance
- Measurement of physical properties
  - density
  - viscosity
  - surface or interfacial tension
  - rheology

5 Sulzer test center in Allschwil, Switzerland.

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