SCALEUP ISSUES FROM BENCH TO PILOT

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ABSTRACT

In the development of commercial processes to convert biomass feedstocks to fuels and chemicals, using batch laboratory results to design and operate a continuous pilot plant presents new challenges. Material handling, solid–liquid separations, stream recycling, and waste stream handling need to be addressed in the pilot plant before the demonstration and commercial plants are built. However, many of these issues are either briefly investigated or completely ignored during laboratory development. Overlooking these potential problems can result in disappointing process performance from the pilot plant, due to mechanical and operating difficulties, and can greatly increase the cost of piloting.

With over 40 years' experience at Hazen Research, Inc. in designing and operating pilot plants for the energy, mining, and environmental fields, the authors will present a number of areas that can present problems in process scaleup from the laboratory to the pilot plant. Many of the problems experienced in pilot operations in other industries have direct application in both biochemical and thermochemical process scaleup. Several examples of piloting problems that can be overcome with proper design and planning are presented. With the knowledge of the potential problems that may be encountered in the pilot plant, the engineer can develop pilot plant designs that will overcome mechanical and operating difficulties, allowing the development team to focus on demonstrating the process and providing design criteria for a feasibility engineering study.

REDUCING RISK IN PROCESS COMMERCIALIZATION

The ultimate goal of spending time and money on a pilot plant is to reduce the risk of commercializing a process. McNulty (1998) evaluated and analyzed 41 case histories of new chemical-based development projects in the mining and inorganic chemical industries to determine the traits of successful and failed projects. The criterion for evaluating each project was to compare the actual plant throughput to the design throughput at various times during plant commissioning. McNulty found that the data could be grouped into four categories, which are averaged in Figure 1.

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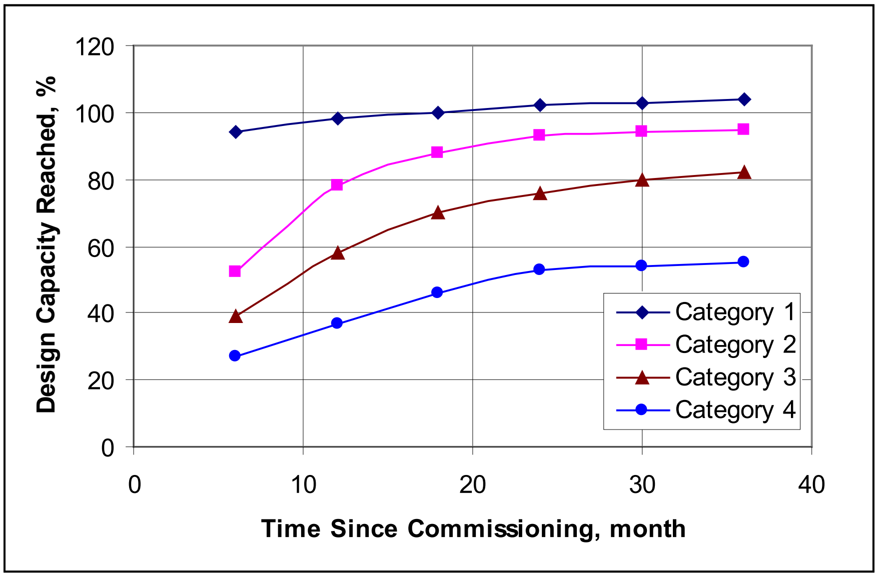

Figure 1. Averaged Plant Production during Commissioning

A close examination of the common characteristics of the 41 projects in this study revealed many similar traits. McNulty summarized these as follows:

Category 1 projects (23 of the 41 evaluated) were the most successful, reaching over 90% nameplate capacity within 6 months and nearly 100% in the first year. Common characteristics included:

- Mature technologies were utilized.
- Equipment was similar in size and duty as that of earlier successful projects.
- Thorough pilot-scale testing of potentially risky unit operations was completed.

Category 2 projects (5 of the 41 evaluated) only achieved about 90% of design capacity, and it took nearly 2 years of commissioning to reach this level. These projects shared at least one and sometimes two or three of the following characteristics:

- The process technology was one of the first.
- Equipment specified for a unit operation was a prototype in terms of size or application.
- Pilot-scale testing was incomplete or was conducted with non-representative samples.
- A key unit operation required unusually severe conditions (high temperature, high pressure, and/or high corrosivity).
- Material handling or intermediate processing had not been carefully engineered.

Category 3 projects (6 of the 41 evaluated) only averaged 80% of design throughput after 2 years and were characterized by issues similar to those of Category 2. In addition, there was also one or more of the following shortcomings:

- Very limited pilot-scale testing was completed.
- Important steps in the process were not addressed during piloting.
- Feed characteristics were not fully understood.
- Product quality was not adequately addressed during process development.
- Engineering, design, and construction were on a “fast track.”

Category 4 projects (7 of the 41 evaluated) shared characteristics similar to those of Categories 2 and 3, but suffered additional problems, including:

- If piloting was conducted, it was for generating product, not confirming process parameters.
- Equipment was downsized or design criteria were made less conservative because of projected cost overruns.
- The process flowsheet was unusually complex, with prototype equipment used in two or more unit operations.
- The process chemistry was not understood.
- Within 36 months of startup, three of the seven Category 4 projects closed.

Based on this analysis, the risks of bringing a successful commercial plant online are greatly reduced by following these guidelines:

- Use commercially available technology whenever possible.
- When developing a process flowsheet in the laboratory, focus on big-ticket steps to reduce costs.
- Carefully plan and execute a pilot program before proceeding to commercialization.

Yes, pilot plants are expensive to build and operate, but changing a process in a full-scale plant will cost much, much more. To quote L.H. Baekeland (the inventor of Bakelite, the first synthetic plastic), “Commit your blunders on a small scale; make your profits on a large scale.”

GOALS OF PILOT PLANT OPERATION

Before proceeding with planning, building, and running a pilot plant, the goals of the pilot plant need to be delineated. Specific goals of a pilot plant operation can include:

- Demonstrating the process on a continuous basis, including yields and product purity.
- Providing design data for scaling the commercial-scale plant.
- Determining the reagent consumptions expected in the commercial plant.
- Providing data for evaluating process economics, including capital and operating costs.
- Obtaining environmental data for permitting.
- Testing materials of construction.
- Determining the potential for scale buildup in processing equipment and methods to minimize scaling.
- Providing product for customer evaluation representative of what will be made in the commercial plant.

PILOT PLANT PLANNING

Once the goals of the pilot plant program have been established, detailed plans for the design and operation can be made, with particular attention to the data that must be collected to design the commercial plant. The authors highly recommend that the engineering firm that will be responsible for building the commercial plant be included in the pilot planning to ensure all of the data needed for the commercial design and operation are gathered during the pilot plant program.

It should be kept in mind that a pilot plant should be a scaled-down version of the commercial operation, not a scaled-up version of the laboratory apparatus. You are trying to avoid the situation illustrated in Figure 2.

Figure 2. What to Avoid during Process Scaleup

There is no rule-of-thumb to help guide the decision about the appropriate size of a pilot plant. There is always a trade-off between keeping the pilot plant small (smaller plants are less expensive to operate, require less feed material, and generate less wastes) and having a large pilot plant that reduces the scaleup factor between the pilot plant and the commercial plant. As noted by Lowenstein (1985), the most important factor is to make certain that the equipment used in the pilot plant is scalable to commercial size. Additionally, the design engineers and outside financing entities may dictate a certain size to increase their comfort level with scaling to commercial size.

Material handling issues can also dictate the minimum pilot size, especially when handling solids. The feed particle size to the process may be fairly large, and the size of the feeding equipment necessary to transport these solids may set the pilot plant size. Small-diameter tubing and piping are much more prone to plugging, so a larger-sized plant with larger-diameter transport lines may be necessary to minimize plugging. Needle valves, tube fittings, and flowmeters tend to easily plug or become fouled at the small scale, so larger sizes and/or alternatives should be considered.

In some instances, one unit operation will require much larger equipment for proper scaleup than the remaining steps. One consideration is to campaign the upstream process to generate feed for the step requiring larger equipment, run the large-scale step, storing the products, and then feed the product or products from this step to the rest of the process. However, a careful examination of the overall process is necessary to determine if this will provide data that are representative of the commercial operation. If the feed or products from the step are prone to alteration from aging, the entire pilot plant may need to be scaled up to match this single step. Also, if recycle streams used in the process affect this step, a larger pilot plant will be necessary.

Another question that arises is what portions of a process should be piloted? Again, there is no hard-and-fast rule; each process needs to be evaluated to determine if all steps should be piloted or if some can be excluded from the pilot plant operation. For instance, off-the-shelf technology, such as distillation of an ethanol–water solution, probably does not need to be piloted. However, if the ethanol or water stream will be recycled back to the process, distillation should be included in the pilot plant operation, so that any adverse effects of impurities in the recycled streams on process performance can be ascertained. Countercurrent decantation (CCD) circuits are used in many commercial operations to settle and wash solids. However, they are extremely difficult to operate at pilot scale. At Hazen Research, we generally separate and wash solids on filters, using wash ratios that simulate CCD operation. To provide the design criteria for the commercial plant, fresh slurry is collected from the pilot operation and bench-scale tests are conducted to provide the required design parameters.

Obtaining Adequate Laboratory Data for the Pilot Plant Design

Obtaining data that are useful for the design of a pilot operation can be more difficult than one might think. Often, primary research used to develop a bench-scale process is performed by researchers who have little or no experience with large-scale operations. Most often it falls to the pilot design engineer to request additional data to adequately address issues that can be expected to arise in pilot operations. It should be remembered that information obtained in the laboratory is much cheaper than obtaining the information in a pilot operation, and it is much cheaper to confirm laboratory data in a pilot plant than in a commercial plant.

It is important to impress upon the primary research team that data collected for scaleup purposes assumes that the core process technology works in ideal conditions, which is to say in a controlled environment being managed by an expert researcher. The data required for the pilot design will often be quite pedestrian in the eyes of the researcher but crucial to the success of the project. Examples of this would be the evaporation characteristics of a recycle stream, the settling behavior of a precipitate, kinetics of crystal formation, or changes in the heat capacity of a reaction mixture over time. An optimal solution is to have a design engineer work directly with the research team to ensure that adequate scaleup data are collected.

The most effective way to reduce uncertainty when scaling a process is to perform the bench-scale research on actual feed material, particularly if the feedstock is known to be subject to seasonal and/or campaign-specific variance. This is especially critical when dealing with biochemical or catalytic processes, in which trace impurities can bring the entire process to a halt.

A good example of this would be a fermentation process, often developed on the basis of a recombinant organism that has been selected to exhibit advantageous behavior. These organisms are often isolated and utilized with clean feedstock (reagent-grade carbohydrate medium), free of impurities that will exist in a commercial operation (wood hydrolyzate). Once the fermentation agent is subjected to actual feedstock, any number of uncertainties can arise. Fermentation inhibition, mutation, and competing microorganisms will all affect the process in a fundamental manner, usually requiring additional primary fermentation or pretreatment research.

Likewise, a number of catalytic processes assume a source of clean H_2 or CO from a gasification step at specific concentration ratios. Often the process is developed with simulated syngas in the lab using reagent-grade bottled gases. As shown in Figure 3 from Kruse (2008), gas composition can vary widely with temperature during gasification, even with a simple feedstock of pure glucose. Kruse reported that when lignin is added to the gasification feedstock in proportions found in nature, H_2, CO, and CH_4 gas yields change significantly under laboratory conditions. Thus the ratios of these gases from actual gasifiers will vary significantly with changing feedstocks and conditions. Gas yields will be subject to additional variance due to the role of water, salts, and proteins. The influence of additional coke and char formation on a catalytic operation introduces further uncertainty.

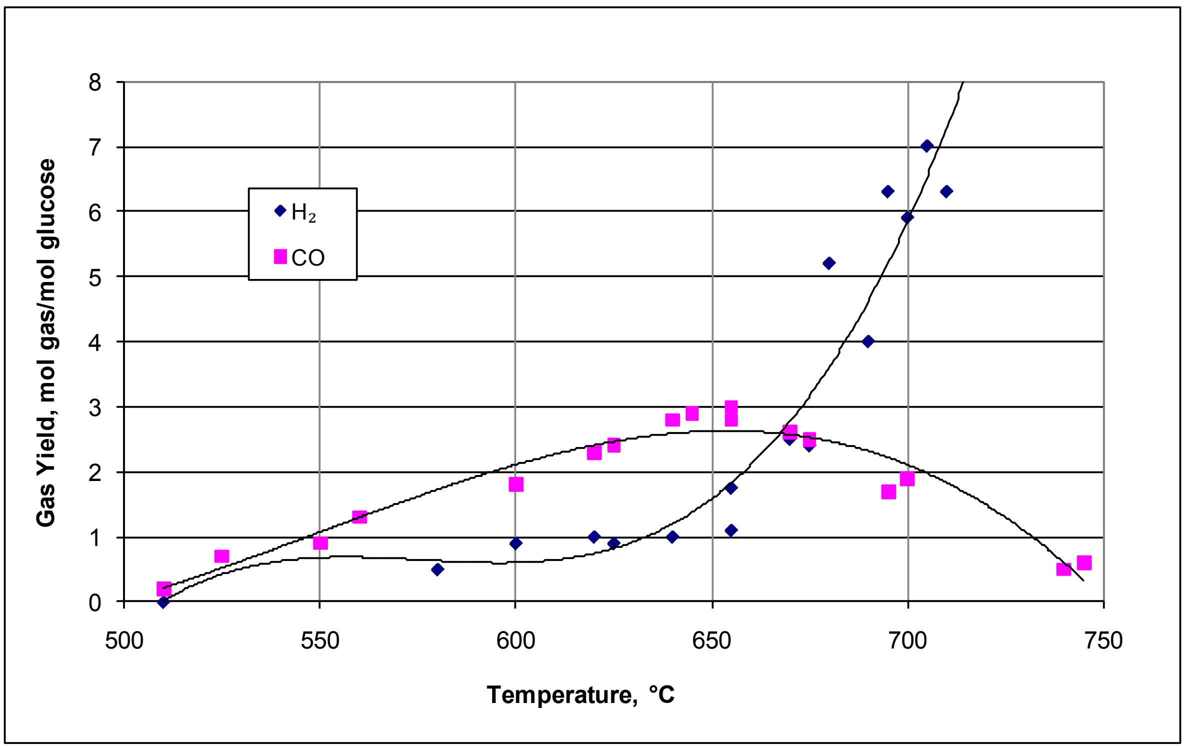

Figure 3. Gas Composition of Hydrothermal Glucose Decomposition as a Function of Temperature (28 MPa, 30 s, 0.6 M glucose)

Basic process development strategies suggest focusing on those process steps that require special equipment or conditions and those processes that offer the potential to reduce commercial costs. This may not be apparent until the design phase of the project because of fundamental scaleup concerns, which are typically not addressed in the primary research effort, but should be included in the scope of the research, even if only to demonstrate that they are not a concern. A few examples would be:

- Heat transfer at larger scale is much more difficult to control than that at bench scale. Large masses (particularly in aqueous processes) are difficult to heat or cool rapidly and will require engineered systems to do so, if it is a requirement of the process. Tube-in-shell heat exchangers are commonly used to accomplish temperature changes and are a robust technology, but are prone to plugging if solids drop from solution during cooling. Likewise, localized superheating can take place, particularly if the flow through a heat exchanger is insufficient.

- As mentioned previously, material handling can be a significant challenge if the process requires a particular particle morphology, size distribution, bulk density, or concentration. Material flow characteristics, reagent hazards, and thermal sensitivity should all be evaluated once the process is lined out.

- Solid–liquid separations are usually the most difficult unit operations at scale and can represent a significant cost in the commercial operation. The process development (from the design engineer's perspective) should focus on minimizing the number of separations that are required. It is a good idea to require the primary research team to use technologies other than a centrifuge during development if at all possible, as they are quite costly to purchase and run. Oftentimes, coagulation, flocculation, or coprecipitation can result in more easily separated slurries. It is important to note that important separation parameters such as the effect of particle agglomeration, cake buildup, compression, and crystal formation may not be determined until sufficient material is available to perform a pilot-scale test. It is useful to combine research batches into a large campaign to generate feed for a test of this sort prior to pilot equipment selection.

- If the process is expected to utilize stream recycling, ensure in the laboratory that the process is not sensitive to variations in stream composition, buildup of impurities, or thermal and chemical breakdown of stream constituents over time. Biochemical processes usually require a significant amount of water removal from process streams. Even if the solution to handle wastewater is just to rely on the local wastewater treatment plant, key process economics in the form of reagents or product can literally go down the drain unless allowances are made for efficient water removal.

- Materials of construction can be easily overlooked. Often economics (and the reality that pilot plants are usually temporary installations) dictate that the pilot equipment be suitable but not optimal for the intended use. In addition to the obvious concern over safety and potential corrosion, it is very possible that reagents can leach impurities from the equipment into the process. Primary researchers are typically familiar with extremely high-quality glassware and reactors and often overlook the potential impact of attempting the process in more mundane vessels. Additionally, pilot-scale equipment will typically require more gasketed connections in process piping. In addition to chemical reactivity, gasket suitability under aggressive conditions may change their suitability over time, as they may deform or degrade.

Ideally, these concerns can be addressed during the technology transfer from the laboratory to the pilot scale. A thorough review of the process flow diagram by the primary research team with input and requests for additional information from the pilot design team will give the best chance of success in the pilot plant.

Solid–Liquid Separation

Solid–liquid separations can be the most expensive unit operations in a commercial operation. Great attention is needed to these steps to minimize the costs for these separations. Spear (2009) states that separation processes (including solid–liquid separations, as well as separating same-phase components from one another) can account for 40–70% of a chemical plant's capital and operating costs.

Kochergin and Miller (2009) point out that biomass properties can be dependent on the harvest time, the growing area, and storage conditions, and that these changing properties may drastically change the filterability of pretreated biomass. This points to the necessity of exploring the variation in process feedstocks in the development program and the critical need for representative biomass samples for the laboratory and pilot plant programs.

Data provided by Kochergin and Miller demonstrate the importance of properly designing the solid–liquid separation step on ethanol yield and the process water balance. They looked at the effect of removing inhibitor compounds on ethanol yield from ammonia-pretreated sugar cane bagasse, shown in Figure 4. In this process, the pretreated biomass is separated from the liquid on a screen, which results in a 5–10% loss of fine biomass through the screen. This biomass loss affects the overall ethanol yield, which is reflected in the three curves in Figure 4.

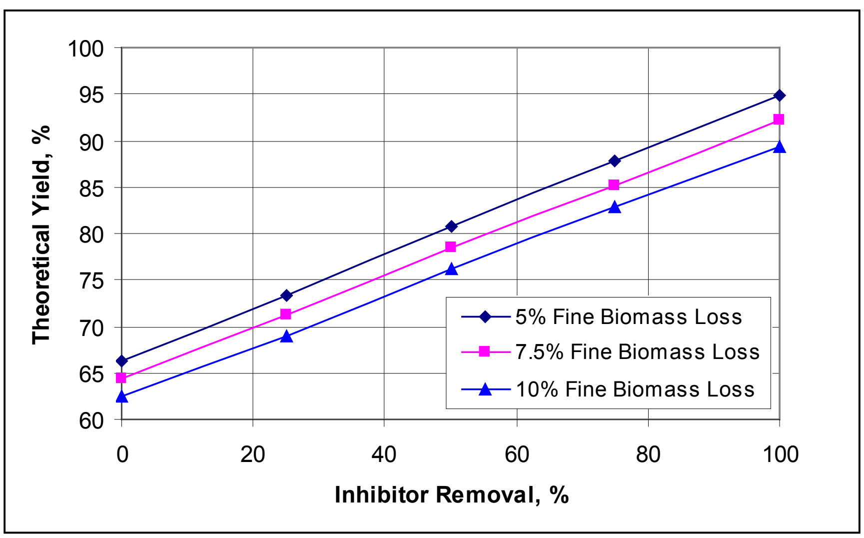

Figure 4. Effect of Inhibitor Removal on Theoretical Ethanol Yield

The removal of inhibitors is directly related to the washing efficiency of the pretreated biomass on the screen. The more water used to wash the biomass, the higher the washing efficiency. Kochergin and Miller show this relationship in Figure 5. Although better wash efficiency results in lower inhibitor concentrations and greater ethanol yields, using more wash water can greatly impact the water balance of the commercial plant, producing much larger wastewater volumes requiring treatment and more dilute streams for downstream processing. It should be remembered that washing efficiencies are also a function of the biomass properties, including the amount of moisture retained by the pretreated biomass. Thus variation in biomass properties will affect the relationship between inhibitor removal and the amount of washing required.

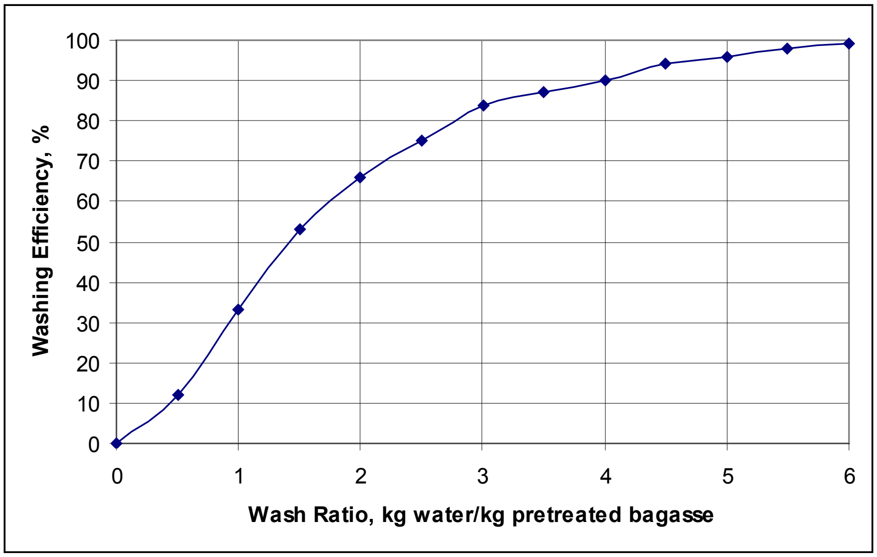

Figure 5. Wash Efficiency as a Function of Washing Ratio

Pilot Plant Safety and Operations

Lastly, the pilot plant must be designed and operated with safety in mind. Just because the pilot plant will be smaller than the commercial operation, safety considerations are no less for pilot plants. In many instances, novel technologies are being scaled up in a pilot plant and potential safety hazards will not be as well-known as for mature technologies.

With proper pilot plant design and execution, and a commercial plant that utilizes the pilot plant results, the pilot plant will accurately predict the performance of the commercial plant. The success in the pilot plant step requires personnel with pilot plant experience to plan and operate the pilot plant and to translate the pilot plant results into design criteria for scaling up to the commercial operation.

CASE STUDIES OF BIOMASS PROCESSING PILOT PLANTS

To illustrate the pitfalls that can be encountered in development programs when scaling from bench-scale to pilot plant operation, three examples are presented by the authors.

Example 1: Dewatering Algae Pond Harvest

The production of biodiesel from algae provides an illustration about the importance of solid–liquid separations in a commercial biofuels process. In the laboratory, researchers routinely concentrate the algal cells by centrifuging the pond harvest to a paste containing 15–25% dry solids. In scaling up the process to commercialization, these researchers assume that industrial-sized centrifuges will be appropriate for this step.

However, a quick calculation for a medium-sized biodiesel operation producing 25,000,000 gal/yr of fuel shows the use of commercial centrifuges is impractical for concentrating pond harvest to 15–25% dry solids. One can calculate that a pond harvest of 320,000 gal/min is necessary for this biodiesel production rate, using these average values provided by Alabi, et al. (2009) and Grima, et al. (2003):

- Pond harvest cell density: 0.1% dry solids (1 g/L)
- Lipid content of the algae: 15% oil
- Biodiesel production from algae oil: 1 gal biodiesel/gal algae oil

Alabi, et al. states that the costs for centrifuging pond harvest to a paste is $1,000–$1,500/dry ton of solids, a cost that is certainly out of the question for a viable economic process.

Most published assessments of solid–liquid separation options consider a two-step concentration process. The first step concentrates the algae solids to 3–5% solids, eliminating 97–98% of the water, and the second step further concentrates the algae solids to 15–25%, depending on the solid–liquid separation technologies and the algal species used.

In surveying potential technologies for the first concentration step, one should review processes used in industries that handle large volumes of water, such as water treatment, wastewater treatment, sewer treatment, and mining operations. The technologies most frequently used for concentrating dilute solids in these industries include flocculation, settling, screening, and dissolved air flotation, or a combination of several of these steps. Flocculation is often considered, especially coupled with another of these technologies, but if chemical flocculants are added, the choice of flocculants must be carefully made. The water recovered from the solid–liquid separation step must be recycled back to algae production, and any residual flocculant in this water cannot hinder algae growth.

Once the algae solids are concentrated to 3–5% solids, a more energy- and capital-intensive separation step can be considered, such a centrifuging or filtering. Because of the small size of the algae cells, filtering is only possible if the solids can be flocculated.

Needless to say, a dedicated laboratory program to determine the most economic solid–liquid separation for concentrating the pond harvest for downstream processing must be completed before proceeding to a pilot plant. If chemical flocculants are used, the dose and cost should be determined during the laboratory work. Recovered water needs to be used to propagate the algae to make sure that cell growth is not adversely affected. Once the concentration steps have been determined in the laboratory, the design parameters for the commercial operation can then be verified during the pilot plant operation.

Example 2: Tar Formation during Biomass Pyrolysis

Thermochemical processes present a unique set of challenges for scaling from the laboratory to piloting and then to commercialization. Key to these processes is removing solids at high temperature and condensing organic tars and liquids from the vapor stream without plugging lines and equipment. The smaller size of a pilot plant compounds this problem because smaller transport lines and the larger equipment surface-area-to-volume ratios allow cold spots to easily develop on which organic tars condense.

Figure 6 shows a photograph of several filter candles used to filter hot solids from the gas stream of a flash pyrolysis pilot plant run at Hazen Research. Because poor heat control allowed cold spots to form, condensing tars comingled with the solids, encrusting the filter candles with a solids–tar mixture that sealed off the filters. Proper temperature control to eliminate cold spots was necessary to overcome this problem.

Figure 6. Filter Candles Encrusted with a Mixture of Tar and Solids

Although coal gasification and pyrolysis have been commercialized for many decades, biomass behaves differently in these thermal processes. The organic vapors contain a different suite of compounds when feeding biomass than when coal is used, with biomass producing tars and oils containing more oxygenated organics. These biomass-derived organics condense at different temperatures than coal-derived organics and can readily polymerize at certain temperatures. Also, the inorganic solids (ash) from biomass have a different elemental composition than coal ash, particularly with respect to alkali metals. This can be particularly troublesome in high-temperature processes because a high alkali metal ash melts at a much lower temperature and can result in fouling by sticky ash. Thus the differences in the physical characteristics between coal and biomass ash requires modification to commercial thermochemical equipment developed for coal.

When designing a thermochemical pilot plant for biomass processing, the offgas treatment system requires careful consideration. The potential for developing cold spots on the solids handling system must be eliminated by proper design to prevent tars from condensing with the solids and plugging the circuit (as illustrated in Figure 6). All lines also need to be designed so no cold spots develop in the circuit. The condensing system to collect tars and oils requires sufficient flexibility so that the condensation temperature can be varied over a wide range. This will provide a robust system to handle differing offgas characteristics as the upstream thermal processing conditions are changed. Startup procedures that eliminate cold spots in the offgas system must be developed. Lastly, the engineer needs to design the pilot plant so that all transport lines, solids removal equipment, and condensers can be easily disassembled and cleaned, because plugging will occur.

Example 3: Base-Catalyzed Degradation Due to Recycled Solvent Use

Biomass feedstocks are also used in the production of specialty chemicals, including pharmaceutical compounds for human and animal use. In this example, the laboratory development work utilized pure solvents. When the process was scaled to the pilot operation, spent solvent was reclaimed by distillation and reused. An impurity was also reclaimed in the distillation column, which contaminated the final pharmaceutical product. This demonstrates the potential problems that can arise when ignoring potential contaminants in recycle streams in a commercial process.

The United States Pharmacopeia–National Formulary (USP–NF) is a book of public pharmacopeial standards, which serves as a guide for companies manufacturing Active Pharmaceutical Ingredients (API). The monograph for active compounds identified specific impurities that are allowed in the purified substance, and at what levels. For a particular API, unless the USP–NF defines an allowable impurity level, the default allowable impurity level is 100 ppm. This applies to all impurities that can be detected in a final API product.

When the feedstock for a particular API is a biomass, the extraction and purification of the API compound normally results in a purified product containing very small amounts of a family of molecules with very similar structures and behaviors. In these instances, the manufacturing process is designed to target removing these specific impurities based on minor chemical differences. The dilemma is always how much of the impurity can be removed from the product without sacrificing yield.

In this example, the process as delivered to the pilot design team used a marker compound to make impurity-based process decisions. Because of the extremely low level of select impurities, the analytical method in use was unable to fully resolve the product impurity profile until almost the last purification step. It was determined at the bench scale that this marker compound would predict the behavior of all impurities roughly 80% of the time. All of the unit operations in the purification process were designed to remove specific product analogues based on polarity, and the marker compound correlation was the basis of a validation of the process.

During the pilot operation of the API process, the first production batch appeared to be within USP–NF specification during in-process testing. At the final purification stage, however, an unknown impurity was detected at levels exceeding the default of 100 ppm. After much consternation and rapid structure elucidation, it was determined that the unknown impurity was a degradation product of a known impurity, and that the process was incapable of removing it. As a result, the pilot batch was destroyed, and the process was sent back to the bench scale to address this new impurity.

It was determined that the unknown impurity was created early in the process due to the buildup of residual weak base in the primary solvent used in the process, which was recycled. All the compounds in the family of the API are subject to base-catalyzed epimerization, and the epimer of the API was an expected impurity; process steps had been designed to accommodate the gradual degradation of the API to its epimer in small amounts. What had not been considered was that some of the key impurities would also undergo this degradation, and these degradants had not been included in the correlation study with the marker compound.

The development of the process at the bench scale had used technical-grade solvent that did not contain enough of the base to affect the epimerization of the impurities. When the switch was made to the recycled solvent at pilot, the process effectively created a new family of impurities that had not been addressed. To overcome this problem, the pilot plant was stopped and the process development program returned to the laboratory to alter the process to handle these impurities. This required that the pilot plant be redesigned, delaying the implementation of the commercial process and greatly increasing the development costs.

CONCLUSIONS

A well-planned pilot plant design and operation is essential to minimize the risk of failure when scaling a process to commercial size. This requires a laboratory development program that not only focuses on the core technology, but also provides the necessary data for a pilot plant to handle separation steps, waste streams, and material handling operations. Additionally, the pilot goals must be clearly stated and the pilot program designed to meet these goals. Although pilot plants are expensive to build and operate, failure to properly plan and execute this step in scaling a process to commercialization can result in lengthy delays in commissioning the commercial plant and even commercial failure.

LITERATURE CITED

Alabi, A. O., Tampier, M., and Bibeau, E. 2009. *Microalgae technologies and processes for biofuels/bioenergy production in British Columbia: Current technology, suitability, and barriers to implementation.* The British Columbia Innovation Council, January 14.

Grima, E. M., Belarbi, E. H., Fernandez, F. G. A., Medina, A. R., and Christi, Y. 2003. Recovery of microalgae biomass and metabolites: Process options and economics. *Biotechnology Advances* 20: 491.

Kochergin, V., and Miller, K. 2009. Evaluation of target efficiencies for solid–liquid separation steps in biofuels production. Paper presented at the 31st Symposium on Biotechnology for Fuels and Chemicals, San Francisco, CA, May 3–6.

Kruse, A. 2008. Supercritical water gasification. *Biofuels, Bioproducts and Biorefining* 2 (5) (September–October): 415.

Lowenstein, J. G. 1985. The pilot plant. *Chemical Engineering* December 9: 62.

McNulty, T. 1998. Innovative technology: Its development and commercialization. In *Managing innovation in the minerals industry*, ed. M. C. Kuhn. Littleton, CO: Society for Mining, Metallurgy, and Exploration.

Spear, M. 2009. Separations in flux. http://www.chemicalprocessing.com/articles/2006/025.html (accessed June 17, 2009).