PROCESS DESIGN

1. The Nature of Chemical Process Design

In a chemical process, the transformation of raw materials into desired chemical products usually cannot be achieved in a single step. Instead, the overall transformation is broken down into a number of steps that provide intermediate transformations. These are carried out through reaction, separation, mixing, heating, cooling, pressure change, particle size reduction or enlargement. Once individual steps have been selected they must be interconnected to carry out the overall transformation (Figure 1**a**). Thus the *synthesis* of a chemical process involves two broad activities. First, individual transformation steps are selected. Second, these individual transformations are interconnected to form a complete process that achieves the required overall transformation. A *flowsheet* is the diagrammatic representation of the process steps with their interconnections.

Once the flowsheet structure has been defined, a *simulation* of the process can be carried out. A simulation is a mathematical model of the process that attempts to predict how the process would behave if it were constructed (Fig. 1**b**). Having created a model of the process, the flowrates, compositions, temperatures, and pressures of the feeds can be assumed. The simulation model then predicts the flowrates, compositions, temperatures, and pressures of the products. It also allows the individual items of equipment in the process to be sized and predicts, eg, how much raw material is being used or how much energy is being consumed. The performance of the design can then be evaluated. There are many facets to the evaluation of performance. Good economic performance is an obvious first criterion, but it is certainly not the only one.

Chemical processes should be designed as part of a sustainable industrial activity that retains the capacity of ecosystems to support both life and industrial activity into the future. Sustainable industrial activity must meet the needs of the present without compromising the needs of future generations. For chemical process design, this means that processes should use raw materials as efficiently as is economic and practicable, both to prevent the production of waste that can be environmentally harmful and to preserve the reserves of raw materials as much as possible. Processes should use as little energy as economic and practicable, both to prevent the build-up of carbon dioxide in the atmosphere from burning fossil fuels and to preserve reserves of fossil fuels. Water must also be consumed in sustainable quantities that do not cause deterioration in the quality of the water source and the long-term quantity of the reserves. Aqueous and atmospheric emissions must not be environmentally harmful, and solid waste to landfill must be avoided.

The process also must meet required health and safety criteria. Start-up, emergency shutdown and ease of control are other important factors. Flexibility, ie, the ability to operate under different conditions such as differences in feedstock and product specification, may be important. Availability, ie, the number of operating hours per year, also may be critically important. Uncertainty in the design, eg, resulting from poor design data or uncertainty in the economic data, might guide the design away from certain options.

Some of these factors, such as economic performance, can be readily quantified, others, such as safety, often cannot. Evaluation of the factors that are not readily quantifiable, the intangibles, requires the judgment of the design team.

Once the basic performance of the design has been evaluated, changes can be made to improve the performance; the process is *optimized*. These changes might involve the synthesis of alternative structures, ie, *structural optimization*. Thus the process is simulated and evaluated again, etc, optimizing the structure. Alternatively, each structure can be subjected to *parameter optimization* by changing operating conditions within that structure.

2. Chemical Products and Process Design Objectives

When considering the design of processes for the manufacture of chemical products, the market into which they are being sold fundamentally influences the objectives and priorities in the design. Chemical products can be divided into three broad classes:

1. *Commodity* or *bulk* chemicals are produced in large volumes and purchased on the basis of chemical composition, purity and price. Examples are sulfuric acid, nitrogen, oxygen, ethylene, and chlorine.
2. *Fine* chemicals are produced in small volumes and purchased on the basis of chemical composition, purity and price. Examples are chloropropylene oxide (used for the manufacture of epoxy resins, ion exchange resins and other products), dimethylformamide (used, eg, as a solvent, reaction medium and intermediate in the manufacture of pharmaceuticals), *n*-butyric acid (used in beverages, flavorings, fragrances, and other products) and barium titanate powder (used for the manufacture of electronic capacitors).
3. *Specialty* or *effect* or *functional* chemicals are purchased because of their effect (or function), rather than their chemical composition. Examples are pharmaceuticals, pesticides, dyestuffs, perfumes, and flavorings.

Commodity chemicals will find use only in industrial processes, whereas some fine and specialty chemicals will find use in consumer products (1). Because commodity and fine chemicals tend to be purchased on the basis of their chemical composition alone, they are *undifferentiated*. For example, there is nothing to choose between 99.9% benzene made by one manufacturer versus another manufacturer, other than price and delivery issues. On the other hand, specialty chemicals tend to be purchased on the basis of their effect, or function, and are therefore *differentiated*. For example, competitive pharmaceutical products are differentiated according to the efficacy of the product, rather than chemical composition. An adhesive is purchased on the basis of its ability to stick things together, rather than its chemical composition, etc.

However, undifferentiated and differentiated should be thought of as relative terms for chemical products, rather than absolute terms. In practice, chemicals tend not to be completely undifferentiated or completely differentiated. Commodity and fine chemical products might have impurity specifications as well as purity specifications. Traces of impurities can in some cases give some differentiation between different manufacturers of commodity and fine chemicals. For example, 99.9% acrylic acid might be considered to be an undifferentiated product. However, traces of impurities, at concentrations of a few parts per million, can interfere with some of the reactions in which it is used and can have important implications for some of its uses. Such impurities might differ between different manufacturing processes. Not all specialty products are differentiated. For example, pharmaceutical products like asprin (acetylsalicylic acid) are undifferentiated. Different manufacturers can produce asprin and there is nothing to choose between these products, other than price and differentiation created through marketing of the product.

Scale of production also differs between the three classes of chemical products. Fine and specialty chemicals tend to be produced in volumes $<1000\ \mathrm{t \cdot year^{-1}}$. On the other hand, commodity chemicals tend to be produced in much higher volumes than this. However, again the distinction is not so clear. Polymers are differentiated products, because they are purchased on the basis of their mechanical properties, but can be produced in quantities significantly $>1000\ \mathrm{t \cdot year^{-1}}$.

When a new chemical product is first developed, it can often be protected by patent in the early years of commercial exploitation. For a product to be eligible to be patented, it must be novel, useful and unobvious. If patent protection can be obtained, this effectively gives the producer a monopoly for commercial exploitation of the product until the patent expires. Patent protection lasts for 20 years from the filing date of the patent. Once the patent expires, competitors can join in and manufacture the product. If competitors cannot wait until the patent expires, then alternative competing products must be developed.

Another way to protect a competitive edge for a new product is to protect it by secrecy. The formula for Coca-Cola has been kept secret for >100 years. Potentially, there is no time limit on such protection. However, for the protection through secrecy to be viable, competitors must not be able to reproduce the product from chemical analysis. This is likely only to be the case for certain classes of specialty products and food products for which the properties of the product depend on both the chemical composition and the method of manufacture.

Figure 2 illustrates different product *life cycles* (2,3). The general trend is that when a new product is introduced into the market, the sales grow slowly until the market is established and then more rapidly once the market is established. If there is patent protection, then competitors will not be able to exploit the same product commercially until the patent expires, when competitors can produce the same product and take market share. It is expected that competitive products will cause sales to diminish later in the product life cycle until sales become so low that a company would be expected to withdraw from the market. In Figure 2, Product A appears to be a poor product that has a short life with low sales volume. It might be that it cannot compete well with other competitive products and alternative products quickly force the company out of that business. However, a low sales volume is not the main criterion to withdraw from the market. It might be that a product with low volume finds a market niche and can be sold for a high value. On the other hand, if it were competing with other products with similar functions in the same market sector, that keeps both the sales price and volume low, then it would seem wise to withdraw from the market. Product *B* in Figure 2 appears to be a better product, showing a longer life cycle and higher sales volume. This has patent protection, but sales decrease rapidly after patent protection is lost, leading to loss of market through competition. A still better product is Product *C* in Figure 2. This shows high sales volume with the life of the product extended through reformulation of the product (2). Finally, Product *D* in Figure 2 shows a product life cycle that is typical of commodity chemicals. Commodity chemicals tend not to exhibit the same kind of life cycles as fine and specialty chemicals. In the early years of the commercial exploitation, the sales volume grows rapidly to a high volume, but then does not decline and enters a mature period of slow growth, or in some exceptional cases slow decline. This is because commodity chemicals tend to have a diverse range of uses. Even though competition might take away some end uses, new end uses are introduced, leading to an extended life cycle.

The different classes of chemical product will have very different *added value* (the difference between the selling price of the product and the purchase cost of raw materials). Commodity chemicals tend to have low added value and are produced in large volumes, whereas fine and specialty chemicals tend to have a high added value and tend to be produced in small volumes.

Because of this, when designing a process for a commodity chemical, it is usually important to keep operating costs as low as possible. The capital cost of the process will tend to be high relative to a process for fine or specialty chemicals because of the scale of production.

When designing a process for specialty chemicals, priority tends to be given to the product, rather than to the process, because the unique function of the product must be protected. The process is likely to be small scale and operating costs tend to be less important than with commodity chemical processes. The capital cost of the process will be low relative to commodity chemical processes because of the scale. The time to market for the product is also likely to be important with specialty chemicals, especially if there is patent protection. If this is the case, then anything that shortens the time from basic research, through product testing, pilot plant studies, process design, construction of the plant to product manufacture will have an important influence on the overall project profitability.

All this means that the priorities in process design are likely to differ significantly, depending on whether a process is being designed for the manufacture of a commodity, fine or specialty chemicals. In commodity chemicals, there is likely to be relatively little product innovation, but intensive process innovation. Also, equipment will be designed for a particular specific process step. On the other hand, the manufacture of fine and specialty chemicals might involve:

- Selling into a market with low volume.
- Short product life cycle.
- A short time to market is demanded, and therefore is less time for process development, with product and process development proceeding simultaneously.

Because of this, the manufacture of fine and specialty chemicals is often carried out in multi-purpose equipment, perhaps with different chemicals being manufactured in the same equipment at different times during the year. The life of the equipment might greatly exceed the life of the product.

The development of pharmaceutical products is such that high quality products must be manufactured during the development of the process to allow safety and clinical studies to be carried out before full-scale production. Pharmaceutical production represents an extreme case for process design in which the regulatory framework controlling production makes it difficult to make process changes, even during the development stage. Even if significant improvements to processes for pharmaceuticals can be suggested, it might not be feasible to implement them, as such changes might prevent or delay the process from being licensed for production.

3. Formulation of the Design Problem

Before a process design can be started, the design problem must be formulated. Formulation of the design problem requires a product specification. If a well-defined chemical product is to be manufactured, then the specification of the product might appear straightforward (eg, a purity specification). However, if a specialty product is to be manufactured, it is the functional properties that are important, rather than the chemical properties, and this might require a *product design* stage in order to specify the product (1,4).

The initial statement of the design problem is often ill-defined. For example, the design team could be asked to expand the production capacity of an existing plant that produces a chemical that is a precursor to a polymer product, which is also produced by the company. This results from an expansion in the demand for the polymer product and the plant producing the precursor currently being operated at its maximum capacity. The design team might well be given a specification for the expansion. For example, the Marketing Department might assess that the market could be expanded by 30% over a 2-year period, which would justify a 30% expansion in the process for the precursor. However, the 30% projection can easily be wrong. The economic environment can change, leading to the projected increase being either too large or too small. It might also be possible to sell the polymer precursor in the market to other manufacturers of the polymer and justify an even larger expansion than 30%. If the polymer precursor can be sold in the market place, is the current purity specification within the company suitable for the market place? Perhaps the market place demands a higher purity than is currently the company specification. Perhaps the current specification is acceptable, but if the specification could be improved, the product could be sold for a higher value and/or a greater volume could be sold. An option might be to not expand the production of the polymer precursor by 30%, but instead to purchase it from the market. If it is purchased from the market, is it likely to be up to the company specifications, or will it need some purification before it is suitable for the company's polymer process? How reliable will the market source be? All of these uncertainties are related more to market supply and demand issues, rather than specific process design issues.

Closer examination of the current process design might lead to the conclusion that the capacity can be expanded by 10% with a very modest capital investment. A further increase to 20% would require a significant capital investment, but an expansion to 30% would require an extremely large capital investment. This opens up further options. Should the plant be expanded by 10% and a market source identified for the balance? Should the plant be expanded to 20% similarly? If a real expansion in the market place is anticipated and expansion to 30% would be very expensive, why not be more aggressive and instead of expanding the existing process, build an entirely new process? If a new process is to be built, then what should be the process technology? New process technology might have been developed since the original plant was built, which enables the same product to be manufactured at a much lower cost. If a new process is to be built, where should it be built? It might make more sense to build it in another country that would allow lower operating costs and the product shipped back to be fed to the existing polymer process. At the same time this might stimulate the development of new markets in other countries, in which case what should be the capacity of the new plant?

From all of these options, the design team must formulate a number of plausible possible design options. Thus from the initial ill-defined problem, the design team must create a series of very specific options and these should then be compared on the basis of a common set of assumptions regarding, for example, raw materials prices and product prices. Having specified an option, this gives the design team a well-defined problem to which the methods of engineering and economic analysis can be applied.

In examining a design option, the design team should start out examining the problem in terms of the overall feasibility with the minimum of detail to ensure the design option is worth progressing (5). Is there a large difference between the value of the product relative to the cost of the raw materials? If the overall feasibility looks attractive, then more detail can be added, the option reevaluated, further detail added, and so on. Byproducts might play a particularly important role in the economics. It might be that the current process produces some byproducts that can be sold in small quantities to the market. But as the process is expanded, there might be market constraints for the new scale of production. If the byproducts cannot be sold, how does this affect the economics?

If the design option appears to be technically and economically feasible, then additional detail can be considered. Material and energy balances can be formulated to give better definition to the inner workings of the process and a more detailed process design developed. The design calculations for this will normally be solved to a high level of precision. However, a high level of precision cannot usually be justified in terms of the operation of the plant after it has been built. The plant will almost never work precisely at its original design flowrates, temperatures, pressures, and compositions. This might be because the raw materials are slightly different than assumed in the design. The physical properties assumed in the calculations might have been in error in some way, or operation at the original design conditions might create corrosion or fouling problems, or perhaps the plant cannot be controlled adequately at the original conditions, etc, for a multitude of other possible reasons. The instrumentation on the plant will not be able to measure the flowrates, temperatures, pressures, and compositions as accurately as the calculations performed. High precision might be required for certain specific parts of the design. For example, the polymer precursor might need certain impurities to be very tightly controlled, perhaps down to the level of ppm. It might be that some contaminant in a waste stream might be exceptionally environmentally harmful and must be extremely well defined in the design calculations.

Even though a high level of precision cannot be justified in many cases in terms of the plant operation, the design calculations will normally be carried out to a reasonably high level of precision. The value of precision in design calculations is that the consistency of the calculations can be checked to allow errors or poor assumptions to be identified. It also allows the design options to be compared on a valid like for like basis.

Because of all the uncertainties when carrying out a design, the specifications are often increased beyond those indicated by the design calculations and the plant *overdesigned*, or *contingency* added, through the application of *safety factors* to the design. For example, the design team might calculate the number of distillation plates required for a distillation separation using elaborate calculations to a high degree of precision only to add an arbitrary extra 10% to the number of plates for contingency. This allows for the feed to the unit not being exactly as specified, errors in the physical properties, upset conditions in the plant, control requirements, etc. If too little contingency is added, the plant might not work. If too much contingency is added, the plant will not only be unnecessarily expensive, but too much overdesign might make the plant difficult to operate and might lead to a less efficient plant. For example, the design team might calculate the size of a heat exchanger and then add in a large contingency and significantly oversize the heat exchanger. The lower fluid velocities encountered by the oversized heat exchanger can cause it to have a poorer performance and to foul up more readily than a smaller heat exchanger. Thus a balance must be made between different risks.

In summary, the original problem posed to process design teams is often ill-defined, even though it might appear to be well defined in the original design specification. The design team must then formulate a series of plausible design options to be screened by the methods of engineering and economic analysis. These design options are formulated into very specific design problems. Some design options might be eliminated early by high level arguments or simple calculations. Others will require more detailed examination. In this way the design team turns the ill-defined problem into a well-defined problem for analysis. To allow for the many unquantifiable uncertainties, overdesign is used. Too little overdesign might lead to the plant not working. Too much overdesign will lead to the plant being unnecessarily expensive, and perhaps difficult to operate and less efficient. A balance must be made between different risks.

Now, consider the basic features of the design of chemical processes.

4. The Hierarchy of Chemical Process Design

Consider the process illustrated in Figure 3 (6). The process requires a reactor to transform the *FEED* into *PRODUCT* (Fig. 3**a**). Unfortunately, not all the *FEED* reacts. Also, part of the *FEED* reacts to form *BYPRODUCT* instead of the desired *PRODUCT*. A separation system is needed to isolate the *PRODUCT* at the required purity. Figure 3**b** shows one possible separation system consisting of two distillation columns. The unreacted *FEED* in Figure 3**b** is recycled, and the *PRODUCT* and *BYPRODUCT* are removed from the process. Figure 3**b** shows a flowsheet in which all heating and cooling is provided by external *utilities* (steam and cooling water in this case). This flowsheet is probably too inefficient in its use of energy, and heat would be recovered. Thus *heat integration* would probably be carried out to exchange heat between those streams that need to be cooled and those that need to be heated. Figure 4 (6) shows two possible designs for the *heat exchanger network*, but many other heat integration arrangements are possible.

The flowsheets shown in Figure 4 feature the same reactor design. It could be useful to explore changes in reactor design. For example, the size of the reactor could be increased to increase the amount of *FEED* that reacts (6). Now there is not only much less *FEED* in the reactor effluent but more *PRODUCT* and *BYPRODUCT*. However, the increase in *BYPRODUCT* is larger than the increase in *PRODUCT*. Thus, although the reactor has the same three components in its effluent as the reactor in Figure 3**a**, there is less *FEED*, more *PRODUCT*, and significantly more *BYPRODUCT*. This change in reactor design generates a different task for the separation system, and it is possible that a separation system different from that shown in Figures 3 and 4 is now appropriate. Figure 5 shows a possible alternative, which also uses two distillation columns, but the separations are carried out in a different order.

Figure 5 shows a flowsheet without any heat integration for the different reactor and separation system. As before, this is probably too inefficient in the use of energy, and heat integration schemes can be explored. Figure 6 (6) shows two of the many possible flowsheets.

Different complete flowsheets can be evaluated by simulation and costing. On this basis, the flowsheet in Figure 4**b** might be more promising than the flowsheets in Figures 4**a**, 6**a**, and **b**. However, the best flowsheet cannot be identified without first optimizing the operating conditions for each. The flowsheet in Figure 6**b** might have greater scope for improvement than in Figure 4**b**, etc.

Thus the complexity of chemical process synthesis is twofold. First, can all possible structures be identified? It might be considered that all the structural options can be found by inspection, at least all of the significant ones. The fact that even long-established processes are still being improved bears evidence to just how difficult this is. Second, can each structure be optimized for a valid comparison? When optimizing the structure, there may be many ways in which each individual task can be performed and many ways in which the individual tasks can be interconnected. This means that the operating conditions for a multitude of structural options must be simulated and optimized.

It is helpful when developing a methodology if there is a clearer picture of the nature of the problem. If the process requires a reactor, this is where the design starts, and is likely to be the only place in the process where raw materials are converted into products. The chosen reactor design produces a mixture of unreacted feed materials, products, and byproducts that need separating. Unreacted feed material is recycled. The reactor design dictates the separation and recycle problem. Thus, design of the separation and recycle system follows reactor design. The reactor and separation and recycle system designs together define the process heating and cooling duties. Thus heat exchanger network design comes next. Those heating and cooling duties that cannot be satisfied by heat recovery dictate the need for external heating and cooling *utilities* (furnace heating, steam use, steam generation, cooling water, air cooling, or refrigeration). Thus utility selection and design follows the design of the heat recovery system. The selection and design of the utilities is made more complex by the fact that the process will most likely operate within the context of a site comprising a number of different processes all connected to a common utility system. The process and the utility system will both need water, eg, for steam generation, and will also produce aqueous effluents that will need to be brought to a suitable quality for discharge. Thus the design of the water and aqueous effluent treatment system comes last. Again, the water and effluent treatment system must be considered at the site level, as well as the process level.

This hierarchy can be represented symbolically by the layers of the "onion diagram" shown in Figure 7 (7). The diagram emphasizes the sequential, or hierarchical, nature of process design. Other ways to represent the hierarchy have also been suggested (5).

Some processes do not require a reactor, eg, some processes just involve separation. Here, the design starts with the separation system and moves outward to the heat exchanger network, utilities, and so on. However, the same basic hierarchy prevails.

The synthesis of the correct structure and the optimization of parameters in the design of the reaction and separation systems are often the single most important tasks of process design. Usually, there are many options, and it is impossible to fully evaluate them unless a complete design is furnished for the "outer layers" of the onion. For example, it is not possible to assess which is better, the basic scheme from Figure 3**b** or that from Figure 5, without fully evaluating all possible designs such as shown in Figures 4**a**, and **b** and 6**a** and **b**, all completed, including utilities.

5. Continuous and Batch Processes

When considering the processes in Figures 3–5 an implicit assumption was made that the processes operated continuously. However, not all processes operate continuously. In a *batch* process, the main steps operate discontinuously. In contrast with a continuous process, a batch process does not deliver its product continuously, but in discrete amounts. This means that heat, mass, temperature, concentration and other properties vary with time. In practice most batch processes are made up of a series of batch and *semicontinuous* steps. A semicontinuous step runs continuously with periodic start-ups and shut-downs.

Consider the simple process shown in Figure 8. Feed material is withdrawn from storage using a pump. The feed material is preheated in a heat exchanger before being fed to a batch reactor. Once the reactor is fully charged, further heating takes place inside the reactor using steam to the reactor jacket, before the reaction proceeds. During the later stages of the reaction, cooling water is applied to the reactor jacket. Once the reaction is complete the reactor product is withdrawn using a pump. The reactor product is cooled in a heat exchanger before going to storage.

The first two steps, pumping for reactor filling and feed preheat are both semicontinuous. The heating inside the reactor, the reaction itself and the cooling using the reactor jacket are all batch. The pumping to empty the reactor and the product cooling step are again semicontinuous.

Many batch processes are designed on the basis of a scale-up from the laboratory, particularly for the manufacture of specialty chemicals. If this is the case, the process development will produce a *recipe* for the manufacturing process. The recipe is not unlike a recipe used in cookery. It is a step-by-step procedure that resembles the laboratory procedure, but scaled to the quantities required for manufacturing. It provides information on the quantities of material to be used in any step in the manufacturing, the conditions of temperature, pressure, etc, at any time, and the times over which the various steps take place. The recipe can be thought of as the equivalent of the material and energy balance in a continuous process. However, care should be taken to avoid taking artificial constraints from the laboratory to the manufacturing process (ie, those constraints imposed by the laboratory procedures that do not apply to industrial plant).

The hierarchy in batch process design is no different from that in continuous processes and the hierarchy illustrated in Figure 7 prevails also for batch processes. However, the time dimension brings constraints that do not present a problem in the design of continuous processes. For example, heat recovery might be considered for the process in Figure 8. The reactor effluent (that requires cooling) could be used to preheat the incoming feed to the reactor (that requires heating). Unfortunately, even if the reactor effluent is at a high enough temperature to allow this, the reactor feed and emptying take place at different times, meaning that this will not be possible without some way to store the heat. Such heat storage is possible but usually uneconomic, especially for small-scale processes.

If a batch process manufactures only a single product, then the equipment can be designed and optimized for that product. The dynamic nature of the process creates additional challenges for design and optimization. It might be that the optimization calls for variations in the conditions during the batch through time, according to some *profile*. For example, the temperature in a batch reactor might need to be increased or decreased as the batch progresses.

Multiproduct batch processes, with a number of different products manufactured in the same equipment, present even bigger challenges for design and optimization (8). Different products will demand different designs, different operating conditions and perhaps different profiles for the operating conditions through time. The design of equipment for multiproduct plants will thus require a compromise to be made across the requirements of a number of different products. The more flexible the equipment, and the configuration of the equipment, the more it will be able to be adapted to the optimum requirements of each product.

Batch processes:

- Are economical for small volumes.
- Are flexible in accommodating changes in product formulation.
- Are flexible in changing production rate by changing the number of batches made in any period in time.
- Allow the use of standardized multipurpose equipment for the production of a variety of products from the same plant.
- Are best if equipment needs regular cleaning because of fouling or needs regular sterilization.
- Are amenable to direct scale-up from the laboratory.
- Allow product identification. Each batch of product can be clearly identified in terms of when it was manufactured, the feeds involved and conditions of processing. This is particularly important in industries such as pharmaceuticals and foodstuffs. If a problem arises with a particular batch, then all of the product from that batch can be identified and withdrawn from the market. Otherwise all of the product available in the market would have to be withdrawn.

One of the major problems with batch processing is batch-to-batch conformity. Minor changes to the operation can mean slight changes in the product from batch to batch. Fine and specialty chemicals are usually manufactured in batch processes. Yet, these products often have very tight tolerances for impurities in the final product and demand batch-to-batch variation being minimized.

6. New Design and Retrofit

There are two situations that can be encountered in chemical process design. The first is in the design of *new plant* or *grassroot* design. In the second, design is carried out to modify an existing plant in *retrofit* or *revamp*. The motivation to retrofit an existing plant could be, eg, to increase capacity, allow for different feed or product specifications, reduce operating costs, improve safety, or reduce environmental emissions. One of the most common motivations is to increase capacity. When carrying out a retrofit, whatever the motivation, it is desirable to try and make as effective use as possible of existing equipment. The basic problem with this is that the design of the existing equipment might not be ideally suited to the new role to which it will be put. On the other hand, if equipment is reused it will avoid unnecessary investment in new equipment, even if it is not ideally suited to the new duty.

When carrying out a retrofit the connections between the items of equipment can be reconfigured, perhaps adding new equipment where necessary. Alternatively, if the existing equipment differs significantly from what is required in the retrofit, then in addition to reconfiguring the connections between the equipment, the equipment itself can also be modified. Generally, the fewer modifications to both the connections and the equipment the better.

The most straightforward design situations are those of grassroot design as it involves the most freedom to choose the design options and the size of equipment. In retrofit, the design must try to work within the constraints of existing equipment. Because of this, the ultimate goal of the retrofit design is often not clear. For example, a design objective might be given to increase the capacity of a plant by 50%. At the existing capacity limit of the plant, at least one item of equipment must be at its maximum capacity. Other items of equipment might be below their maximum capacity. The differences between the spare capacity of different items of equipment in the existing design arises from errors in the original design data, different design allowances (or *contingency*) in the original design, changes to the operation of the plant relative to the original design, etc. An item of equipment at its maximum capacity is the *bottleneck* to prevent increased capacity. Thus, to overcome the bottleneck or *debottleneck*, the item of equipment is modified, or replaced with new equipment with increased capacity, or a new item is placed in parallel or series with the existing item, or the connections between existing equipment reconfigured, or a combination of actions. As the capacity of the plant is increased from the existing limit, then different items of equipment will limit. Thus there will be thresholds in the plant capacity created by the limits in different items of equipment. To overcome each threshold requires all equipment with capacity less than the threshold to be modified in some way, or the plant reconfigured, to overcome the threshold. To overcome each threshold requires capital investment. As capacity is increased from the existing capacity limit, ultimately it is likely that the investment to overcome one of the design thresholds will be prohibitive. This is likely to become the design limit, as opposed to the original remit of a 50% increase in capacity in the example above.

In batch processes, the production steps are connected together through time to form a production schedule. At any instant, equipment may be in use or may be idle. As production is maximized, the production schedule will create a bottleneck, as a result of some process step in the schedule becoming the limiting step. Debottlenecking batch processes must therefore consider both the schedule and the equipment design simultaneously (8).

7. Approaches to Chemical Process Design

In broad terms, there are two approaches to chemical process design:

1. *Creating an irreducible structure*. The first approach follows the "onion logic", starting the design by choosing a reactor and then moving outward by adding a separation and recycle system, etc. At each layer, decisions must be made based on the information available at that stage. The ability to look ahead to the completed design might lead to different decisions. Unfortunately, this is not possible, and instead, decisions must be based on an incomplete picture.

   This approach to creation of the design is one of making a series of best local decisions, which might be based on the use of *heuristics* or *rules of thumb* developed from experience (5) or a more systematic approach.

Equipment is added only if it can be justified economically on the basis of the information available, albeit an incomplete picture. This keeps the structure *irreducible*, and features that are technically or economically redundant are not included.

There are two drawbacks to this approach:

a. Different decisions are possible at each stage of the design. To be sure the best decisions have been made, the other options must be evaluated. However, each option cannot be evaluated properly without completing the design for that option and optimizing the operating conditions. This means that many designs must be completed and optimized in order to find the best.
b. Completing and evaluating many options gives no guarantee of ultimately finding the best possible design, as the search is not exhaustive. Also, complex interactions can occur between different parts of a flowsheet. The effort to keep the system simple and to not add features in the early stages of design may result in missing the benefit of interactions between different parts of the flowsheet in a more complex system.

The main advantage of this approach is that the design team can keep control of the basic decisions and interact as the design develops. By staying in control of the basic decisions, the intangibles of the design (eg, safety) can be included in the decision making.

2. *Creating and optimizing a superstructure*. In this approach a *reducible* structure, known as a *superstructure*, is first created that has embedded within it all feasible process operations and all feasible interconnections that are candidates for an optimal design structure. Initially, redundant features are built into the superstructure. As an example, consider Figure 9 (9), which shows one possible structure of a process for the manufacture of benzene from the reaction between toluene and hydrogen. In Figure 9, the hydrogen enters the process with a small amount of methane as an impurity. Thus in Figure 9, the option is embedded of either purifying the hydrogen feed with a membrane or passing it directly to the process. The hydrogen and toluene are mixed and preheated to reaction temperature. Only a furnace has been considered feasible in this case because of the high temperature required. Then two alternative reactor options, isothermal and adiabatic reactors, are embedded, and so on. Redundant features have been included in an effort to ensure that all features that could be part of an optimal solution have been included.

The design problem is next formulated as a mathematical model. Some of the design features are continuous, describing the operation of each unit (eg, flowrate, composition, temperature, and pressure), its size (eg, volume and heat-transfer area) as well as the costs or profits associated with the units. Other features are discrete (eg, a connection in the flowsheet is included or not, a membrane separator is included or not). Once the problem is formulated mathematically, its solution is carried out through implementation of an optimization algorithm. An *objective function* is maximized or minimized (eg, profit is maximized or cost is minimized) in a *structural* *and parameter* optimization. The optimization justifies the existence of structural features and deletes those features from the structure that cannot be justified economically. In this way the structure is reduced in complexity. At the same time, the operating conditions and equipment sizes are also optimized. In effect, the discrete decision-making aspects of process design are replaced by a discrete/continuous optimization. Thus the initial structure in Figure 9 is optimized to reduce the structure to the final design shown in Figure 10 (9). In Figure 10, the membrane separator on the hydrogen feed has been removed by optimization, as has the isothermal reactor and many other features of the initial structure shown in Figure 9.

There are a number of difficulties associated with this approach:

a. The approach will fail to find the optimal structure if the initial structure does not have the optimal structure embedded somewhere within it. The more options included, the more likely it will be that the optimal structure has been included.

b. If the individual unit operations are represented accurately, the resulting mathematical model could be extremely large and the objective function that must to be optimized extremely irregular (10). The profile of the objective function can be like the terrain in a range of mountains with many peaks and valleys. If the objective function is to be maximized (eg, maximize profit), each peak in the mountain range represents a *local optimum* in the objective function. The highest peak represents the *global optimum*. Optimization requires searching around the mountains in a thick fog to find the highest peak, without the benefit of a map and only a compass to tell direction and an altimeter to show height. On reaching the top of any peak, there is no way of knowing whether it is the highest peak because of the fog. All peaks must be searched to find the highest. There are crevasses to fall into that might be impossible to climb out of.

   Such problems can be overcome in a number of ways. The first way is by changing the model such that the solution space becomes more regular, making the optimization simpler. This most often means simplifying the mathematical model. A second way is by repeating the search many times, but starting each new search from a different initial location. A third way, exploits mathematical transformations and bounding techniques for some forms of mathematical expression to allow the global optimum to be found (11).

   A fourth way is by allowing the optimization to search the solution space in a series of discrete moves that initially allow the possibility of going down hill, away from an optimum point, as well as uphill. As the search proceeds, the ability of the algorithm to move downhill must be gradually taken away. Such search strategies that allow deterioration in the objective function in the early stages of optimization require a *stochastic optimization* strategy (12,13).

c. The most serious drawback of this approach is that the design engineer is removed from the decision making. Thus, the many intangibles in design, such as safety and layout, that are difficult to include in the mathematical formulation, cannot be taken into account satisfactorily.

On the other hand, this approach has a number of advantages. Many different design options can be considered at the same time. Also, the entire design procedure can be automated and is capable of producing designs quickly and efficiently.

In summary, the two general approaches to chemical process design of building an irreducible structure and creating and optimizing a superstructure both have advantages and disadvantages. The complex multiple trade-offs usually encountered in chemical process design can be handled by this approach. However, whichever is used in practice, there is no substitute for understanding the problem.

8. Process Simulation

Having created a process structure in a flowsheet, the design requires the material and energy balance to be evaluated with greater accuracy. In turn, this will allow a preliminary sizing of equipment and a more detailed economic evaluation of the design. Computer simulation packages are normally used to evaluate the material and energy balance once the basic process structure has been established.

To understand how such computer packages function, consider the simple flowsheet in Figure 11**a**. This involves an isomerization of Components A to B. The mixture of A and B from the reactor is separated into relatively pure A, which is recycled, and relatively pure B, which is the product. No byproducts are formed.

To solve the material and energy balance requires a series of material and energy balance equations can be written for the flowsheet. There are two basic approaches used to solve the equations.

- *Equation-orientated* The *equation-orientated* or *equation-based* approach solves the set of equations simultaneously. Whilst this approach seems straightforward for a simple mass balance, for more complex recycle systems with energy balance equations and phase equilibrium equations, it is not straightforward. Equations describing the flowsheet connectivity are combined with equations describing the various unit operations in the flowsheet and, if possible, the physical property correlations into one large equation set (8). The solution of the set of equations can be performed by a general purpose nonlinear equation solver. Because of the difficulties of including the physical property equations, these are often formulated as distinct procedures and kept separate from equations describing the flowsheet connectivity and unit operations (8).
- *Sequential Modular* In the *sequential modular* approach, the process equations are grouped within *unit operation blocks*. Each unit operation block contains the equations that relate the outlet stream and the

performance variables for the block to the inlet stream variables and specified parameters. Each unit operation block is then solved one at a time in sequence (8). The output calculated from each block becomes the feed to the next block, etc. Figure 11**b** shows the block structure for the flowsheet in Figure 11**a**. The direction of information flow usually follows that of the material flow. First, the feed stream must be created. This feed then goes to a mixer where the fresh feed is mixed with the recycle stream. Here a problem is encountered, as the flowrate and composition of the recycle are unknown. The sequential modular solution technique is to *tear* one of the streams in the recycle loop. In Figure 11**b**, the recycle stream itself has been torn. In general, tearing the recycle stream itself is only one option for tearing a stream in a loop. It is often best to tear a stream for which a good initial estimate can be provided. Tearing determines those streams or information flows that must be torn to render the system (or subsystem) to be acyclic. A *recycle convergence unit* or *solver* is inserted in the tear stream, Figure 11**b**. To start the calculation of the material balance in Figure 11**b**, values for the component molar flowrates for the recycle stream (tear stream) must be estimated. This allows the material balance in the reactor and separator to be solved. In turn, this allows the molar flowrates for the recycle stream to be calculated. The calculated and estimated values can then be compared to test whether errors are within a specified tolerance.

If a material balance is to be solved then the convergence variables can be taken to be the component molar flowrates. When a material and energy balance is to be solved, the additional convergence variables are usually taken to be pressure and enthalpy.

The equation-oriented approach and the sequential modular approach each have their relative advantages and disadvantages. The sequential modular approach is intuitive and easy to understand. It allows the design engineer to interact with the solution as it develops and errors tend to be more straightforward to understand than with the equation-oriented approach. However, large problems may be difficult to converge with the sequential modular approach. On the other hand, the equation-oriented approach can make it difficult to diagnose errors. It is generally not as robust as the sequential modular approach and generally requires a good intialization to solve. One major advantage of the equation-oriented approach is the ability to formulate the problem as an optimization problem, as design problems almost invariably involve some optimization.

Of course, the two approaches can be combined and the sequential modular approach used to provide an initialization for the equation-oriented approach.

9. Process Control

Once the basic process configuration has been fixed, a *control system* must be added. The control system compensates for the influence of external *disturbances*, such as changes in feed flowrate, feed conditions, feed costs, product demand, product specifications, product prices, ambient temperature, etc. Ensuring safe operation is the most important task of a control system, which is achieved by monitoring the process conditions and maintaining them within safe operating limits. While maintaining the operation within safe operating limits, the control system should optimize the process performance under the influence of external disturbances. This involves maintaining product specifications, meeting production targets and making efficient use of raw materials and utilities.

A control mechanism is introduced that makes changes to the process in order to cancel out the negative impact of disturbances. In order to achieve this, instruments must be installed in order to measure the operational performance of the plant. These *measured variables* could include temperature, pressure, flowrate, composition, level, pH, density, and particle size. *Primary measurements* may be made to directly represent the control objectives (eg, measuring the composition that needs to be controlled). If the control objectives are not measurable, then *secondary measurements* of other variables must be made and these secondary measurements related to the control objective. Having measured the variables that need to be controlled, other variables need to be *manipulated* in order to achieve the control objectives. A control system is then designed, which responds to variations in the measured variables and manipulates variables to control the process.

Industrial practice is to first design and optimize the process configuration (if necessary taking into account multiple states) and then to add the control system. However, there is no guarantee that design decisions made on the basis of steady-state conditions will not lead to control problems once process dynamics are considered. For example, an item of equipment might be oversized for contingency, because of uncertainty in design data or future debottlenecking prospects, based on steady-state considerations. Once the process dynamics are considered, this oversized equipment might make the process difficult to control, because of the large inventory of process materials in the oversized equipment. The approach to process control should adopt an approach that considers the control of the whole process, rather than just the control of the individual process steps in isolation (14).

As further details of the instrumentation, control and auxiliary pipes, valves, and storage required for the final engineering design are added, the *flowsheet* or *process flow diagram* develops into the *piping and instrumentation diagram* (*P &I D*).

10. Process Safety

Early decisions made purely for process reasons can often lead to safety problems that require complex solutions later in the development of the design. It is far better to consider safety problems early as the design progresses, even though it has been considered last here for the sake of explanation. Designs that avoid the need for hazardous materials, or use less of them, or use them at lower temperatures and pressures, or dilute them with inert materials will be *inherently safe* and will not require elaborate safety systems (15).

The best way of dealing with a hazard in a flowsheet is to remove it completely. The provision of safety systems to control the hazard is much less satisfactory. One of the principal approaches to making a process inherently safe is to limit the inventory of hazardous material, *intensification* of hazardous material. The inventories to be avoided most of all are flashing, flammable liquids or flashing, toxic liquids. It may be possible to synthesize flowsheets that do not require large inventories of materials in the process.

If possible, hazardous materials should be used under less hazardous conditions of less extreme temperatures or pressures, or as a vapor rather than superheated liquid, or diluted, in other words *attenuation* (15).

Once the details of the piping and instrumentation diagram have been developed, a detailed *hazard and operability* study can be carried out. This study should follow a structured approach to examining the safety consequences of abnormal circumstances in the process.

11. Chemical Process Design: Summary

The original design problem posed to the design team is often ill-defined, even if it appears on the surface to be well defined. The design team must formulate well-defined design options from the original ill-defined problem and these must be compared on the basis of consistent criteria.

Chemical products can be divided into three broad classes: commodity, fine, and specialty chemicals. Commodity chemicals are manufactured in large volumes with low added value. Fine and specialty chemicals tend to be manufactured in low volumes with high added value. The priorities in the design of processes for the manufacture of the three classes of chemical products will differ.

The design might be a new design or the retrofit of an existing process. If the design is a retrofit, then one of the objectives should be to maximize the use of existing equipment, even if it is not ideally suited to its new purpose.

Both continuous and batch process operation can be used. Batch processes are generally preferred for small scale and specialty chemicals production.

Design starts at the reactor. The reactor design dictates the separation and recycle problem. Together, the reactor design and separation and recycle dictate the heating and cooling duties for the heat exchanger network. Those duties that cannot be satisfied by heat recovery dictate the need for external heating and cooling utilities. The process and the utility system both have a demand for water and create aqueous effluents, giving rise to the water system. This hierarchy is represented by the layers in the “onion diagram“, Figure 7. Both continuous and batch process design follow this hierarchy, even though the time dimension in batch processes brings additional constraints in process design.

There are two general approaches to chemical process design:

1. Creating an irreducible structure.
2. Creating and optimizing a superstructure.

Both of these approaches have advantages and disadvantages.

12. Acknowledgment

Large sections of this article have been taken from R. Smith, *Chemical Process Design and Integration*, 2005 with permission of John Wiley & Sons, Ltd.

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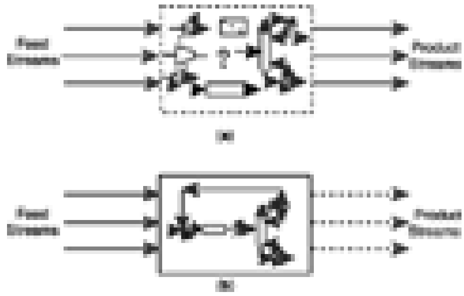

Fig. 1. Synthesis is the creation of a process to transform feed streams into product streams. Simulation predicts how it would behave if it was constructed. (**a**) Process design starts with the synthesis of a process to convert raw materials into desired products. (**b**) Simulation predicts how a process would behave if it was constructed.

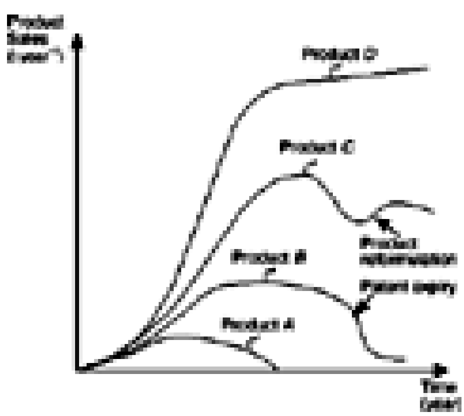

Fig. 2. Product life cycles. (Adapted from P.N. Sharratt, 1997. Handbook of Batch Process Design, Blackie Academic and Professional by permission.)

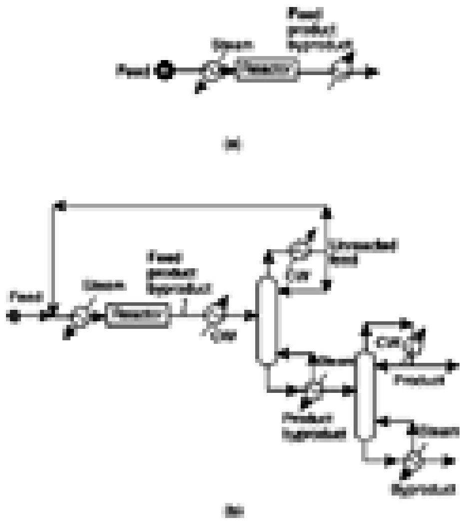

Fig. 3. Process design starts with the reactor. The reactor design dictates the separation and recycle problem. (From R. Smith and B. Linnhoff, 1998. *Trans IChemE ChERD*, 66:195 by permission of the Institution of Chemical Engineers.) (**a**) A reactor transforms feed into product and byproduct. (**b**) To isolate the product and recycle unreacted feed we need a separation system.

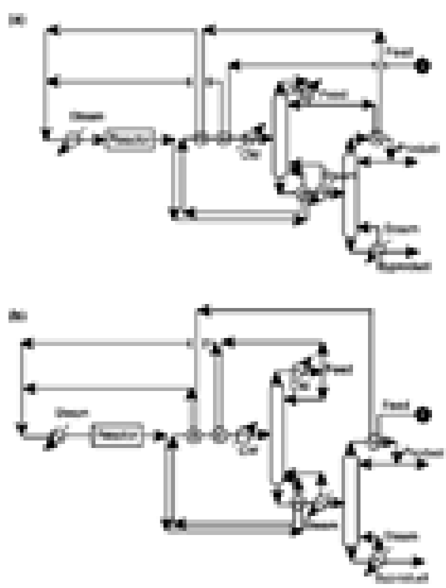

Fig. 4. For a given reactor and separator design there are different possibilities for heat integration. (From R. Smith and B. Linnhoff, 1998. *Trans IChemE ChERD*, 66:195 by permission of the Institution of Chemical Engineers.)

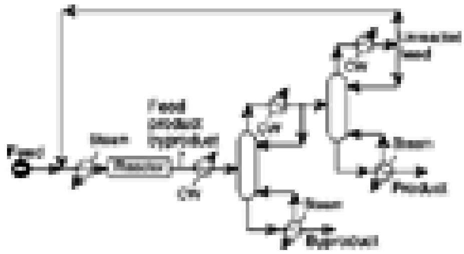

Fig. 5. Changing the reactor dictates a different separation and recycle problem. (From R. Smith and B. Linnhoff, 1998. *Trans IChemE ChERD*, 66:195 by permission of the Institution of Chemical Engineers.)

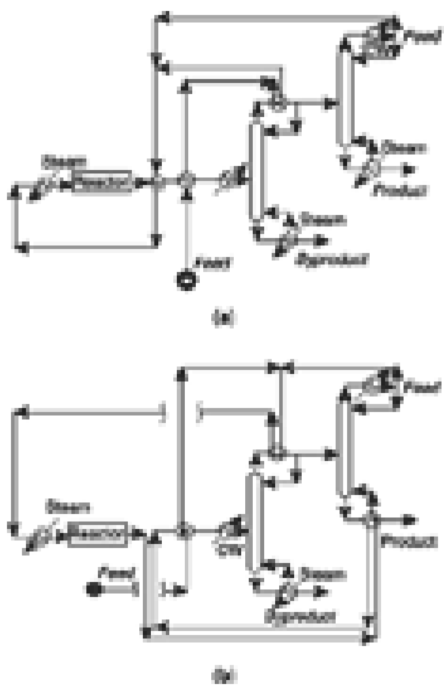

Fig. 6. A different reactor design not only leads to a different separation system but additional possibilities for heat integration. (From R. Smith and B. Linnhoff, 1998. *Trans IChemE ChERD*, 66:195 by permission of the Institution of Chemical Engineers.)

Fig. 7. The onion model of process design. A reactor is needed before the separation and recycle system can be designed, etc.

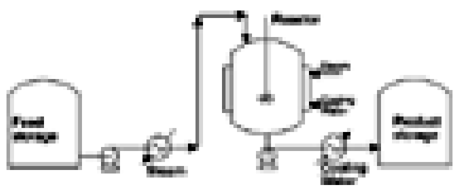

Fig. 8. A simple batch process.

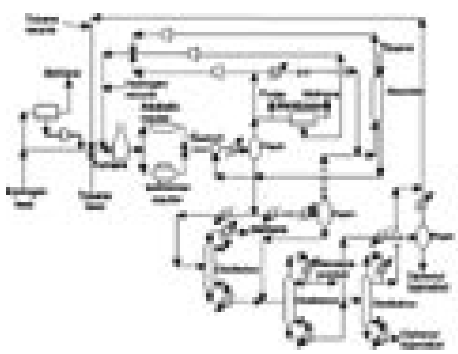

Fig. 9. A superstructure for the manufacture of benzene from toluene and hydrogen incorporating some redundant features. (From G.R. Kocis and I.E. Grossman, 1998, *Comp Chem Eng*, **13**: 797, reproduced by permission.)

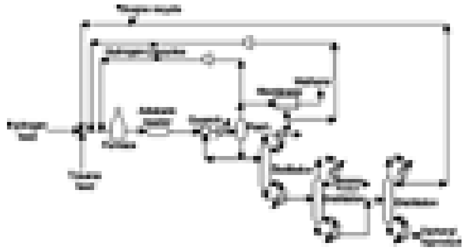

Fig. 10. Optimization discards many structural features leaving an optimised structure. (From G.R. Kocis and I.E. Grossman, 1998, *Comp Chem Eng*, **13**: 797, reproduced by permission.)

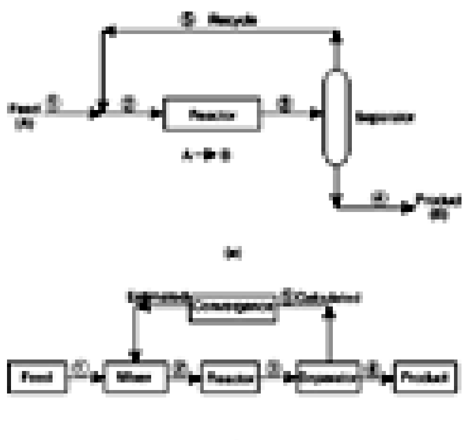

Fig. 11. A simple process with recycle. (**a**) Process flowsheet. (**b**) Block structure of sequential modular calculation.