Separations: Perspective of a Process Developer/Designer

Rakesh Agrawal
Air Products and Chemicals, Inc., Allentown, PA 18195

Introduction

Separation processes play an essential role in the chemical process industries (CPI) for purifying raw materials, recovering product streams of desired purity and preventing pollution through treatment of waste streams released to the environment. Separation processes generally require substantial expenditures for both capital and energy. Depending on the chemical process, the investments in separations and supporting facilities can typically account for 30% to 70% of the total capital cost. Processes operate at feed rates ranging over a factor of 10^{12}—from a few milligrams per hour to several million kilograms per hour.

Separation processes span a diverse range of applications. The National Research Council (1987) report on Separation and Purification lists more than 50 different types of separation processes. Important industries using separation processes include commodity and specialty chemicals, fertilizers, pulp and paper, biotechnology/pharmaceutical, microelectronics, food processing, plastics, metals, and drinking water. The major separation processes are based on absorption, adsorption, crystallization, distillation, extraction, evaporation, filtration, ion exchange, and permeation (membrane, etc.).

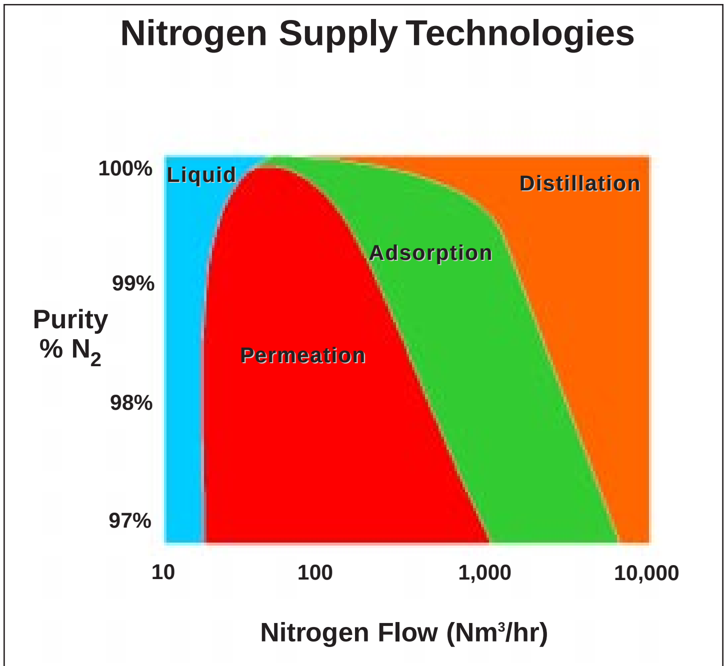

Figure 1. Different separation technologies for nitrogen production.

Given this large scope, we are not going to focus on any specific process here. Instead, we will discuss some of the emerging trends and their effects on separation processes. The forces impacting separation processes are the same as those influencing CPI in general: intense global competition, stringent environmental regulations, increasing raw material and energy costs, customer demand for better quality control, and the need for safe and flexible operating plants. In today's competitive climate, the developer of a separation process not only has to come up with a better solution at a cost that is lower than the prevailing cost, but the time available to accomplish these goals continues to shrink. This "better, cheaper, faster" trend also affects the manner in which separation processes are chosen and designed by practicing engineers. Taken together, the forces mentioned above are leading to:

- Utilization of multiple separation technologies for certain applications.
- Innovations at small scale.
- Increased use of integrated separation and hybrid processes.
- Improvements in the energy-efficiency of separation processes.
- Creation of separation methods for ultrahigh purity chemicals.
- Development of separation processes for the management of waste streams.

Each of these elements is discussed below.

Utilization of multiple separation technologies for certain applications

Quite often a separation task can be achieved by more than one technology. Therefore, in certain applications, multiple technologies may be used depending on product purity and production level. Historically, the developers of one technology have overstated the benefits and concentrated on the shortcomings of the technology that they wished to displace. While competition is healthy for the development of any technology, a practicing designer must be impartial and understand the pros and cons of all the competing technologies. For example, in a recent Perspective article, Rekoske (2001) presented alternative separation technologies available for chiral processes for separating optically pure chemical compounds in the pharmaceutical industry. Some of the separation technologies available for this application are crystallization, kinetic resolution, reaction/resolution combinations, membranes and liquid chromatography. Because some of these technologies are still undergoing significant development, designers of a chiral separation processes must keep track of all these developments and choose the best one available.

A classic example of coexistence of multiple separation technologies is the recovery of nitrogen and oxygen from air. Figure 1 shows the currently preferred separation technologies as a function of the purity of the nitrogen and the production rate. Such a situation leads to a continual reduction in product cost, because the competition between the different technologies leads to innovation in each one.

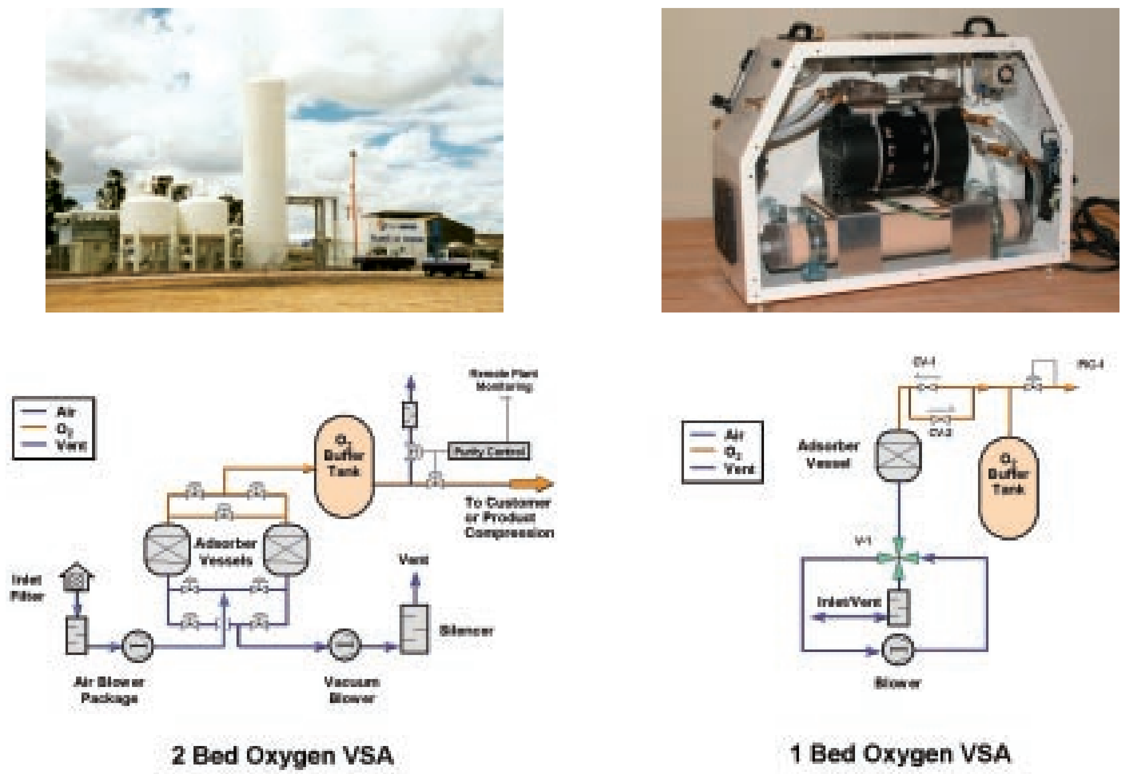

Figure 2. Oxygen vacuum swing adsorption process and plants.
Left: Two-bed process for 10,000–40,000 L/min oxygen; Right: Much simplified and novel one-bed process for 5 L/min oxygen.

Innovations at Small Scale

It is widely believed that as production rate is increased, the cost per unit of product is decreased, due to improved "economies of scale". This has led to the ever-increasing size of chemical plants and their associated separation processes. When the products of these large facilities are used at many widely separated locations, however, distribution costs may become as important as production costs. Smaller plants have advantages as well. They include increased safety (by avoiding the need for storage of large quantities of chemicals) and improved portability of the production facilities. This has led to the recent trend to build much smaller units right at the use location. However, smaller units cannot simply be reduced-size versions of their larger cousins; they are often quite different processes, containing fewer items of more novel equipment. These small separation plants can sometimes be almost as efficient as the larger plants, so the final delivered product costs can be quite comparable. Some of these small new plants require a very high level of innovation on the part of the separation process developer. They also must be very robust, as frequent maintenance is generally not feasible. These objectives are generally met by simplifying the separation unit, as well as its support system to its core, and eliminating any unnecessary equipment. Designing low-capital, power-efficient, lightweight plants requires a thorough understanding, not only of separation unit operation, but also of its interaction with support equipment.

An example of such innovation is seen in the production of 90–95% oxygen by adsorption technology. Several plants in the size range of 10,000–40,000 L/min of oxygen flow using two-bed vacuum swing adsorption are in operation (Figure 2). While several improvements have been made in the past to push the application of this technology to the upper end of production, the use for a much smaller-size range was thought to be uneconomical, in competition with vaporized liquid oxygen. However, innovative improvements—using only single adsorption bed, one four-way valve, and a blower that works as a compressor for half of the cycle and as a vacuum blower for the other half of the cycle—have made this technology economically attractive at a production rate as low as 5 L/min! Figure 2 shows such a unit designed and built by engineers at Air

Products and Chemicals. This “plug-and-play” unit is safer, contains less equipment, and is lower in energy consumption than the existing pressure swing adsorption process that adsorbs at a couple of atmospheres pressures and requires several pressure vessels, etc.

Another interesting example of how a small-size application is leading to cheaper and more efficient systems through innovative integration of separation processes with other operating units is in the production of hydrogen for fuel cell applications. Engineers at Tokyo Gas in Japan have succeeded in developing a sophisticated hydrogen reformer/separator for incorporation into a Polymer Electrolyte Fuel Cell (PEFC) for residential applications. To achieve conventional economies of scale, hydrogen plants with production rates exceeding 100,000 Nm^3/h are being built for refinery applications. Therefore, it is quite remarkable that Tokyo Gas has been able to build the compact unit shown in Figure 3 that produces less than 1 Nm^3/h of hydrogen for 1 KW-class PEFC systems. This unit cleverly integrates the well-known steps of steam generation, catalytic reforming, shift conversion, and CO removal to produce hydrogen for the fuel cell. We expect to see many such innovative examples of portable systems for point-of-use applications.

Figure 3. 1 kW fuel cell incorporating hydrogen generation system.
Courtesy: Tokyo Gas, Japan

Increased Use of Integrated Separation and Hybrid Processes

The driving forces mentioned earlier are demanding cheaper and more energy-efficient plants. One route to these goals is through the use of more compact process equipment. For example, the equipment for one separation process may be integrated with that for another type of separation process, or with another unit operation. This has been termed “process intensification,” a concept that challenges the normal sequential approach to unit operation design. Stankiewicz and Moulijn (2000) provide several examples of process intensification that incorporate separation processes. The device in Figure 3 is an example of process intensification.

A widely-recognized example of an industrial-scale integrated separation process is Eastman Chemical’s process for the production of methyl acetate. By using an innovative single-column reactive-extractive distillation design, both the capital and operating costs were reduced to just one-fifth of those for the optimized, conventionally designed flowsheet (Siirola, 1995). Malone and Doherty (2000) provide a nice commentary on the potential of reactive distillation. There are also several examples where other separation processes such as membrane and adsorption have been integrated with reaction. The ion transport membrane (ITM) synthesis gas process, which is currently under development, has the potential to dramatically reduce capital investment for natural gas-to-liquid plants, and for small-scale distributed hydrogen units. In this unit, the feed air is passed on one side of the membrane and the permeating oxygen is consumed on the other side through autothermal reforming of natural gas. The resulting ceramic membrane reactors are very light and compact (Dyer et al., 2000).

Hybrid separation processes using two or more separation techniques will become more widely used. It will be essential, however, that the separation technologies used are truly “complementary” and that they do not add unnecessarily to capital cost or operating complexity. Conversely, if a separation can be achieved with reasonable economics by a single separation technique, it would be extremely difficult to justify the use of an additional technology. This generally requires that the complementary separation technologies perform a separation that cannot be done by either one alone or that the use of one eliminates significant equipment from the other separation process. There are several examples where hybrid separation processes have successfully reduced the overall product cost without increased capital investment (Stankiewicz and Moulijn, 2000). One specific example is the recovery of volatile organic compounds (VOCs) from a gaseous stream using membrane or adsorption along with a chiller for partial condensation. Another example is the recovery of hydrogen produced in a gasifier or reformer using a membrane followed by a cryogenic partial condensation unit (Agrawal et al., 1988). Here, the use of a membrane at the warm end eliminates the need for the expensive cryogenic expanders and enables the production of product streams of several purities. In general, requirements for high purity products, together with the simultaneous production of other product streams of lower purity, favor hybrid separation processes that truly are improvements over simpler processes.

Improvement in the Energy-Efficiency of Separation Processes

Increased energy cost and stricter environmental regulations will make it necessary to build more energy-efficient separation processes. People around the world are becoming increasingly sensitive to the emission of greenhouse gases. The debate over the Kyoto protocol has further contributed to this global awareness. Any successor to this protocol would undoubtedly also limit CO_2 emissions. The environmental regulations needed to achieve this result will inevitably put pressure on the chemical industry to reduce its energy consumption. Approximately 43% of the energy used in the chemical and petroleum industries is consumed by separation processes (Humphrey et al., 1991).

It is natural to expect that the newer separation technologies can undergo significant improvements in energy efficiency. However, it is quite remarkable to see the tremendous progress that has recently been made for "mature" technologies, such as distillation. According to one estimate, there are 40,000 distillation columns in operation in the U.S. CPI. Many of these columns can be displaced by the use of more efficient processes, and others can be retrofitted with improved distillation devices. The development of low-pressure drop structured packing, for example, is making important contributions to increased distillation efficiency. For multicomponent distillation, a fully thermally coupled distillation column configuration not only needs just one reboiler and one condenser, but often has a substantially reduced heat demand as well. For distillation into three product streams, a divided wall column version of fully thermally coupled distillation is gaining popularity (Lestak and Collins, 1997). Because the divided wall arrangement replaces several columns with a single distillation column, it can be quite cost-effective. Use of this arrangement has recently been made possible through innovations in design and control. Divided wall single-column arrangements are being explored for the distillation of feed mixtures containing more than three components into separate component streams. We have recently learned how to draw fully thermally coupled distillation column arrangements using multiple distillation columns with only one reboiler and one condenser, which are more flexible and easier to operate (Agrawal and Fidkowski, 1998). We are also learning about double-effect and multi-effect multicomponent distillation schemes, which have the potential to reduce the already low heat demands of thermally coupled distillation schemes by 20 to 50% (Siirola, 1995; Agrawal, 2000). It is especially noteworthy that each of the energy saving arrangements described above is accompanied by decreased capital investment!

Efficiency gains are also being achieved in batch distillation. The increased demand for specialty chemicals, which are generally manufactured in smaller quantities, require batch distillation columns which are more flexible and allow rapid turnaround. Several recent studies have made progress towards the understanding of a batch distillation column with multiple holdup vessels (Skogestad et al., 1997). Improved understanding of operating dynamics has the potential to substantially shorten overall cycle time for a run campaign, leading to a major reduction in energy requirements. The ability to distill more batches in a given amount of time can also lead to lower capital investment.

Creation of Separation Methods for Ultrahigh Purity Chemicals

There is an increasing demand for a large array of chemicals with ever-increasing purity. Examples include the pharmaceutical industry and the emerging semiconductor- and nanotechnology-based industries. In the semiconductor industry, the purity specification of chemicals seems to be limited only by the detection limit of the analytical methods. For example, the level of impurities such as H_2, CO, O_2, etc., in nitrogen is often specified to be below 10 parts per billion ("eight nines"). Nitrogen with "six nines" purity—once considered "clean"—has become "dirty" for the semiconductor industry! Not only must the concentration of unwanted, major constituents be reduced to extremely low levels, but also those of minor components that are present in air at ppm levels. The removal of such dilute level impurities to produce ultrahigh purity nitrogen and oxygen requires multiple separation technologies. Currently, novel separation processes using adsorption, catalytic oxidation, and multicomponent distillation provide the needed separations. A typical semiconductor fabrication site requires several dozen ultrahigh purity chemicals. Some of these are quite hazardous or corrosive, and demand special handling procedures. Production of these chemicals has generally been met by the development of novel separation methods using multiple separation techniques. Design of these processes requires engineers with expertise in multiple separation technologies.

Development of Separation Processes for the Management of Waste Streams

According to a U.S. Department of Energy (2000) report, the U.S. chemical industry produced 450 million tons of hazardous waste in 1989, which represented over 90% of the total amount of hazardous waste generated by the U.S. manufacturing segment. In recent years, the chemical industry has made an effort to lessen the environmental impact of chemical production by investing billions of dollars for pollution abatement and control. However, due to increasingly stringent environmental regulations, it is becoming necessary to continue the development of improved pollution abatement processes. Whether at the source, or at the end-of-pipe, improved separation processes will play a dominant role. For example, a number of separation processes based on adsorption or membrane technologies are commercially available. Release of VOCs into the air is a regulated emission. Membrane, adsorption, condensation, or their hybrid processes can be used for VOC control. Selection of a process depends on the concentration of VOCs in the air and the volumetric flow of air (Koros, 1995). Novel, monolithic, and adsorbent wheels ranging in diameters from about 0.2 m to at least 4.2 m are available for VOC removal (Keller, 1995). We will continue to see the development of such innovative processes and technologies to purify air and water with less energy consumption and at a much lower capital cost.

Conclusion

Recent market and environmental forces require the rapid development of better and cheaper separation process solutions. Successful implementation requires a broad view. Often, more than one separation technology can successfully perform a given task. It is necessary not only to choose the "right" solution among the available separation technologies, but also to generate creative and novel solutions by integrating one separation technology with other separation technologies or with other unit operations. We are finding that a more integrated, task-oriented approach to separation to be more beneficial than the traditional, sequential approach. Chemical engineers with expertise in diverse separation technologies and their support systems are creating integrated separation processes at all levels of production and product purity. Clearly, the separation developer/designer with in-depth expertise in more than one separation technology, and a good working knowledge of other separation technologies, will continue to be most effective.

The "mantra" of better and cheaper often means lower capital investment and more energy-efficient processes at all production sizes—from small units for point-of-use—to large, world-scale, industrial units. In recent times we have seen very substantial improvements in energy-efficiency for all the major separation processes. Creative and innovative solutions have even been suggested and implemented for very "mature" technologies such as distillation. Because market and other forces are providing impetus for creativity and innovation across the entire spectrum of separation technologies, these are exciting times for separation process developers and designers.

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