ChE curriculum

# TEACHING SEPARATIONS Why, What, When, and How?

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S eparation costs often control the profitability of plants. In most chemical, petroleum, petrochemical, and pharmaceutical plants, separations account for 40% to 70% of both capital and operating expenses.<sup>[11]</sup> Separations are also the basis for many businesses that involve the manufacture of adsorption systems, distillation columns, extractors, membrane equipment, etc. Finally, separations/mass transfer and reaction engineering represent the two areas that are uniquely "owned" by chemical engineering. Thus, every chemical engineer should have a background in separations.

Unfortunately, beyond acknowledging the importance of separations the consensus evaporates. Few curricula can cover all of the separation techniques used commercially. Every industry specializes in different separations—mechanical separations such as filtration and settling; classical equilibrium-separations, including distillation, absorption, crystallization, and extraction; newer membrane techniques including gas permeation, reverse osmosis, ultrafiltration, and pervaporation; and various adsorption and ion-exchange techniques. And there are even newer processes, such as supercritical fluid extraction, that are finding their way to industrial usefulness.

Ideally, we would first decide what to teach and then decide when to teach it. But often this does not occur because of competing demands on the curriculum. We may select a



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Finally, there is the question of how to teach separations. I recommend an eclectic approach that includes classical graphical and analytical methods, computer simulations, and laboratory experience.

#### WHAT TO TEACH

If most of the graduates of a program were to go into a single industry, the needs of that industry could be used to answer the "what to teach" question. We can classify industries by the separations they use. The petroleum industry is a heavy user of distillation plus absorption, extraction, and separation of two liquid phases. Petrochemicals would probably add membrane separations and adsorption to this list. Pharmaceutical companies are much more interested in centrifugation, filtration, membrane separators, extraction, solution crystallization, precipitation, and chromatography, while inorganic chemical production often relies heavily on solution crystallization and filtration. The commercial gas industry uses cryogenic distillation, membranes, adsorption, and some absorption, and the production of high-purity water uses distillation, membranes, and ion exchange. Fine chemicals may use all of the above processes plus molecular distillation and melt crystallization. Food processing would add drying and freeze drying to the list, while environmental applications often add various types of mechanical separators such as cyclones, decanters, electrostatic precipitators, magnetic separators, and sedimentation. So, it is evident that the mix of jobs taken by graduates of most chemical engineering programs is much too large to choose what to teach based solely on the hiring industry.

A number of schemes have been devised to classify sepa-

#### TABLE 1 **Classification of Separations Based on Similarities in Analysis** Classical Equilibrium-Staged . Distillation, absorption, stripping, extraction, leaching, crystallization from solution Advanced Classical . Extractive distillation, azeotropic distillation · Melt crystallization · Batch distillation Rate-Based . Membrane: gas permeation, pervaporation, ultrafiltration, reverse osmosis, nanofiltration, dialysis, electrodialysis Molecular distillation · CSD in crystallization from solution · Adsorption: thermal and pressure swing, desorbent and purge Ion exchange Chromatographic: column and simulated moving bed

- Mechanical Centrifuge, cyclone, decanter, demister, expression, electrostatic separator, filtration, flotation, magnetic, high-gradient magnetic separator, sedimentation, sink-float
  - New Supercritical extraction
    - Liquid membrane
      - Electrophoresis

# TABLE 2 Chemical Engineering Courses in Ideal Separations Curriculum

Sophomore	Mass and Energy Balances	Thermodynamics I
Junior	Thermodyamics II. Equilibrium Equilibrium-Staged Separations Fluid Dynamics	Heat/Mass Transfer Kinetics/Reactor Design Solids/Mechanical Separations ChE Laboratory I
Senior	Rate Separation Processes Process Dynamics/Control ChE Laboratory II ChE Elective	ChE Design ChE Elective
Possible Separation Electives	Advanced Equilibrium-Staged Separations Azeotropic, extractive and reactive distillation Multicomponent batch distillation and extraction Supercritical extraction	
	Novel and Unusual Separations Clathration Electrophoresis, isoelectric j Liquid membranes Molecular distillation Sublimation/desublimation	focusing, and isotachophoresis

ration methods.<sup>[2-5]</sup> A simple classification based on similarities in the theories is shown in Table 1. The classical equilibrium-staged processes can be operated in staged equipment such as columns and can be analyzed with a stage-by-stage calculation procedure, assuming each stage is in equilibrium. The equilibrium assumption eliminates the need for a mass transfer analysis. Of course, these systems can be operated in different equipment such as random and structured packings, and different analysis procedures can be used. The advanced classical methods can also be analyzed by a stage-by-stage calculation, but with added complexity. Extractive and azeotropic distillation add a third component that makes application of graphical methods difficult; melt crystallization involves movement of solid, and batch distillation adds time as a variable.

Rate-based separations require a mass-transfer analysis. Membrane separations are probably the easiest of the ratebased separations to teach since they often operate at steadystate. Although molecular distillation is not difficult to teach, it is usually ignored. Crystallization from solution appears in Table 1 twice because the concentration of products can be calculated from equilibrium considerations, but the very important crystal size distribution (CSD) requires population balances. Adsorption, ion exchange, and chromatography are the most difficult to teach since they are rate-based and are usually operated as time-dependent processes.

Mechanical separations are involved with the separation of bulk phases. Thus, the mechanisms of separation are inherently different than the diffusional mechanism of equilibrium-staged and rate processes. It has been common to cover the mechanical separations in a fluids course, in a separate course on solids processing, or not at all. Unfortunately, "mechanical separations are grossly under-represented in the typical curriculum relative to industrial practice."<sup>[6]</sup>

The newer processes can be moved into the equilibriumstaged (supercritical extraction) and rate-based (liquid membranes and electrophoresis) portions of the table if educational materials are available. Only supercritical extraction, however, seems to have enough industrial application to justify its inclusion in an undergraduate course.

An alternate approach is to pick a "typical" process in the field being studied and to list all of the separations employed in the process. These are then studied in their proper order. This approach has been most commonly employed in bioseparations.<sup>[7,8]</sup> With this approach, one covers some separations from the mechanical, rate-based, and equilibrium separations in Table 1. The advantage of the approach is relevance; the disadvantage can be lack of depth.

Ideally, undergraduates would study most of the separations in Table 1 in depth. This would require three courses equilibrium-staged operations, rate-based separations, and mechanical separations. I believe industry would like the resulting *Ideal Separations Curriculum* (Table 2). Compared to many chemical engineering curricula, this curriculum is light in transport and design courses and may have one extra ChE course. Unfortunately, the separation experts have been unable to convince their faculty colleagues that separation is more important than everything else in the curriculum!

# ... there is the question of how to teach separations. I recommend an eclectic approach that includes classical graphical and analytical methods, computer simulations, and laboratory experience.

The old compromise curriculum used equilibrium-staged separations with a heavy dose of distillation as *the* "separations course." Mechanical separations were touched on in fluids courses and in lab. Rate-processes occasionally appeared in design courses, but the students really did not know how to design them. Dual-level electives might be available on advanced classical processes, rate-based separations, and solids processing. Details for an equilibrium-staged course and a rate-based separations course are given by Wankat, *et al.*<sup>[5]</sup> At a time when almost all chemical engineering graduates went into the petroleum and chemical process industries, this compromise made sense. "Distillation is used to make 90-95% of all separations in the chemical process industry."<sup>[1]</sup>

Since the ideal separations curriculum is unlikely to happen, what is an appropriate compromise for current times when chemical engineers work in such a wide variety of industries? I suggest the following: the "separations course" should cover the classical equilibrium separations in both staged and packed columns (~80%) and membrane separations (~20%). These topics fit together because the separations tend to be complementary in process plants and the pedagogical difficulty is about the same. A modest amount of mechanical separations (filtration and sedimentation) should be included in the fluids course, and laboratory and design classes should include a variety of separations from the three major categories in Table 1. Dual-level electives should be available in both rate-based separations and solids processing for those students who want more depth.

### WHEN TO TEACH SEPARATIONS

Some chemical engineering curricula teach separations in the sophomore year, some in the junior year, and some in the senior year. Equilibrium-staged courses have an advantage in that they can be taught at any time after the basic mass and energy balance course. They can be taught before mass transfer if this will help balance the curriculum. An interesting experiment and book combined equilibrium-staged separations processes with mass and energy balances in a sophomore course.<sup>[9]</sup> This book was not widely adopted, perhaps because it required changing well-established curricula. A more common place for equilibrium-staged separations is in

the junior year after thermodynamics and either before or at the same time as mass transfer.

A few programs have put the main separations course in the senior year, but it is more common to have laboratory and design courses that require separations as a prerequisite in the senior year. The senior year is the most common time for technical electives.

We also teach separations to graduate students. This used to be the course where students learned computer calculations for multicomponent distil-

lation, absorption, and extraction, but with the advent of powerful simulators these topics have been moved into the undergraduate curriculum. Graduate school is the ideal time to teach adsorption and chromatography, which are conceptually more difficult than most of the other separations. Ideally, schools would make an advanced separations course a core requirement in the graduate program. This would be particularly appropriate in a nonthesis masters programs since it mainly educates students for industrial careers. One possible core course would start with an overview of equilibrium and rates separations and then focus on either adsorption and chromatography or mechanical separations. An alternative is a course focusing on modern analyses of advanced distillation topics, including azeotropic, extractive, and reactive distillation.<sup>[10]</sup> A third, more general, alternative is an advanced mass transport course with applications in separations and other areas.

## HOW TO TEACH SEPARATIONS

How to teach separations may be the most interesting question of all, and of course, it interacts with the why, what, and when. The classical method of teaching separations was to use graphical methods, including McCabe-Thiele, Ponchon-Savarit, and triangular diagrams. Lectures were complemented by laboratory operation of distillation, adsorption, extraction, and evaporation equipment, sometimes of fairly large scale. As computers became readily available, however, the graphical approaches were supplemented with assignments to write FORTRAN code for distillation columns. Graphical methods have the advantage of helping students visualize the separation, but they no longer represent the modern practice of chemical engineering.

Modern chemical engineering practice to design and simulate equilibrium-staged separations to a large extent involves using commercial process simulators such as AspenPlus, ChemCad, Hysys, and Prosim. To be prepared for commercial practice, students need experience simulating and designing equilibrium-staged separations using a commercial simulator. Although the simulators have differences, the one that is used is probably not important (the companies that sell simulation packages may disagree). Unfortunately, students often treat simulators as black-boxes and tend to believe the results they obtain without further checking. Thus, fundamentals and hand calculations (graphical or with a calculator) should still be required. It is also useful to require students to repeat a simulation with different equilibrium correlations. Trying to explain the differences in their results will convince many students that the choice of VLE or LLE correlations is critically important. Suddenly, thermodynamics is relevant!

I believe that graphical methods still have an important place in the curriculum since they foster visualization and serve as a thinking tool. Practicing engineers commonly use McCabe-Thiele diagrams to understand or help debug simulation results. Modern tools such as simulators or spreadsheets<sup>[11]</sup> can be used to draw accurate graphical solutions and thus remove the tedium associated with graphical solutions. Laboratory experience is still necessary since simulators need data, and simulations do not always match reality.

Best-practice principles for teaching should, of course, be employed.<sup>[12]</sup> Introductory courses need to be structured to lead from inductive to deductive reasoning. Start with simple, specific examples and build to more complex cases, then generalize and develop an abstract understanding of the analysis. Finally, deductively show how other separations can be designed using similar techniques (see Haile<sup>[13]</sup> for a complete description of this procedure). Reviews of previously studied techniques can be done deductively. Be sure students actively process material.

Simulators can be incorporated into lecture courses by scheduling a computer lab that meets approximately every other week. Essentially no class time needs to be spent training students to use steady-state simulators. With a good set of instructions and help from the laboratory teaching assistants, the students can become proficient with the separation parts of the simulator while they solve problems. The same simulator should be used throughout the curriculum. Students will then obtain more practice solving separation problems in their laboratory and capstone design courses.

Simulators are beginning to be used extensively in industry for the design of rate-based separations. The adsorption and chromatography simulators are powerful, but quite complicated. In a few years these simulation packages should be used in graduate courses on rate-based separations.

#### SUMMARY AND CONCLUSIONS

The questions in the title have been answered as follows:

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## Why?

Because separations have overwhelming economic significance and they are at the core of chemical engineering.

### What?

All students should study the classical equilibrium-staged processes and receive an introduction to a rate-based process such as membrane separators. Required courses or electives should be available in rate-based separations and mechanical separations. Different separation experiments should be available in laboratory, and separation should be an integral part of senior design projects. Graduate students should study adsorption and chromatography or mechanical separations.

#### When?

The required course should be taught when it fits into the curriculum, which is often the junior year. Electives are normally taught in the senior year. A graduate core course in separations is recommended.

#### How?

Lecture courses should be integrated with a computer laboratory for practice with a modern process simulator. Introductory courses should follow an inductive pattern. Both graphical and analytical methods should be included. The course(s) in separations should be reinforced with separations laboratory experiments and design projects that include separations.

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