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Publisher *Taylor & Francis*

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Separation & Purification Reviews

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713597294>

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Online publication date: 07 October 2000

To cite this Article King, C. Judson(2000) 'FROM UNIT OPERATIONS TO SEPARATION PROCESSES', Separation & Purification Reviews, 29: 2, 233 — 245

To link to this Article: DOI: 10.1081/SPM-100100010

URL: <http://dx.doi.org/10.1081/SPM-100100010>

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FROM UNIT OPERATIONS TO SEPARATION PROCESSES¹

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ABSTRACT

The Arthur D. Little concept of unit operations embodied a number of different methods of separating mixtures and represented a major advance in chemical engineering. Over time, those and subsequent concepts have evolved into a unified field of separation processes. The ways in which this happened are traced. The more unified view of separations enables more coherent and powerful approaches for process selection and design, reducing energy requirements, for selecting separating agents, understanding the complex interactions of mass transfer and phase equilibria, and identifying new methods for separating complex mixtures. As such, separation processes provide one of the most effective vehicles for teaching and understanding the *engineering* of chemical processes.

INTRODUCTION

It is common nowadays to recognize separations (or separation processes, or separations technology) as a distinct field within chemical engineering. This

¹ Invited presentation at the Annual Meeting, American Institute of Chemical Engineers, Miami Beach FL—November 16, 1998.

observation is borne out by the existence of textbooks, university courses and scientific and professional meetings on the subject, a formal Separations Division within the American Institute of Chemical Engineers, two Separations Subdivisions within the American Chemical Society, and designated separate sections for reports of separations research in both the *AIChE Journal* and *Industrial and Engineering Chemistry Research*.

Such was not the case thirty and more years ago. The concept of unit operations subsumed a number of methods of separation and a variety of other processing methods. Separations were not considered as a field unto themselves. The story of the original development of the unit operations concept and the evolution of a major portion of it to the field of separation processes is an interesting one that is essentially coincident with what is now the 90+-year history of the American Institute of Chemical Engineers.

The genesis of a unified field of separation processes came about as the unit operations were categorized, as the fields of mass transfer and transport phenomena were developed, as it became recognized that common methods of analysis (or variants upon them) applied to a number of different methods of separation, and as commercial separations needs and methodologies became more numerous and varied. This transition achieved major impetus from two less obvious sources, as well. These were the intense focus upon isotope-separation processes and other methods of separation during the Manhattan Project of World War II, and growing attention to separations within chemistry and analytical chemistry, in particular the growth of chromatography and the development of membrane materials.

THE UNIT OPERATIONS CONCEPT

The origin of the concept of unit operations is described by Brown et al. (1950), as follows:

“Although the importance of . . . operations that are common to different industries was recognized as early as 1893 [now 107 years ago] by Professor George Lunge [of the Eidgenössische Technische Hochschule (ETH), Zürich], the concept of unit operations was first crystallized by A[rthur] D. Little in 1915 [in a report of the Visiting Committee for the Department of Chemistry and Chemical Engineering to the President of M. I. T., as follows].”

‘Any chemical process, on whatever scale conducted, may be resolved into a coordinate series of what may be termed “Unit Operations,” as pulverizing, drying, roasting, crystallizing, filtering, evaporating, electrolyzing, and so on. The number of these basic operations is not large and relatively few of them are involved in any particular process. The complexity of chemical engineering results from the variety of condi-

tions as to temperature, pressure, etc., under which the unit operations must be carried out in different processes, and from the limitations as to materials of construction and design of apparatus imposed by the physical and chemical character of the reacting substances.’’

By Little’s definition, the Unit Operations are a variety of generic building blocks that can be assembled in largely sequential fashion to compose processes for any of a large variety of applications. Considerable efficiency is achieved in process design and in education by recognizing that the natures of the individual building blocks are much the same in different applications, except that operating conditions and details of equipment design will, of course, vary. The development of the unit-operations concept was in many ways the simplification and generalization that launched the profession of chemical engineering on its way.

In these earlier days, the concept of unit operations contrasted with and complemented the concept of unit processes, which are individual processes useful for making a variety of products (Groggins, 1935).

THE EVOLUTION TOWARD MASS TRANSFER AND MASS-TRANSFER OPERATIONS

It is probably easiest to trace the evolution of the unit-operations concepts by considering the succession of relevant textbooks. The text out of M. I. T. by Walker, Lewis and McAdams called “Principles of Chemical Engineering” was first published in 1923. It was the first widely used book to cover the subject of unit operations, and dealt with them from Little’s point of view. The book considered a wide variety of operations, including fluid flow, heat exchange, combustion, furnaces and kilns, gas producers, crushing and grinding, filtration, evaporation, distillation, absorption, extraction, humidification, air conditioning and drying. Six of these fourteen operations are, in fact, separations. The third edition of Walker, Lewis, McAdams and Gilliland (1937) was the text upon which I cut my teeth in chemical engineering as a student at Yale in the 1950s.

Another classical unit operations text was the one authored by George Granger Brown and numerous co-authors from the University of Michigan, published in 1950. They organized the unit operations by the phases of matter handled—first solids handling, then fluids handling, then the concept of staged operations, which of course included a number of types of separations, and finally heat and mass transfer. What was, in effect, a follow-up to this book was the text by Foust and several co-authors from Lehigh (Foust, et al, 1962). It differentiated between staged operations and continuous-contact operations, which they called “rate” operations, with principles of mass, heat and momentum transfer developed as a basis for analysis of the “rate” operations. General analyses of stage and rate operations were followed by sequential considerations of the

equipment aspects of individual unit operations. The long-running text by McCabe and Smith (1956, 1967, 1976), later joined by Harriott (1985, 1993), continued the approach of dealing with unit operations individually, but grouped them by fluid mechanics, heat transfer and its applications, mass transfer and its applications, and operations involving particulate solids. These three texts took the step of clearly dividing among those unit operations based upon fluid flow, heat transfer and mass transfer.

In his book “Mass Transfer Operations”, Treybal (1955, 1968, 1980) separated out the mass-transfer-based operations and covered them alone. Since separation processes based upon phase equilibration—the most common type—necessarily involve the transfer of matter between phases, these mass-transfer operations are very largely equilibration separation processes, but not exclusively so; e. g., humidification was included. Treybal covered diffusion and mass transfer, then gas-liquid operations, liquid-liquid operations, and solid-fluid operations, with chapters on individual operations within these categories. More recently, Sherwood, Pigford and Wilke (1975) covered the more fundamental aspects of mass transfer as well as some of the same operational aspects.

CASCADE THEORY AND RELATED CONCEPTS

At this point in the story, we need to bring in two quite different lines of development. The first of these is the development of cascade theory and the means of understanding, designing and analyzing separations having separation factors close to unity. The Manhattan Project of World War II brought urgent and major needs for isotope separation, notably production of highly enriched U^{235} and production of deuterium. Because of the extreme staging and energy needs for these separations, it became important to develop new methods of separation, to screen reliably on a theoretical basis among candidate separation processes, and to obtain the most workable and energy-efficient designs for the processes selected. Karl Cohen of General Electric Company (Cohen, 1951), Manson Benedict of Kellogg Corporation and others undertook the development of what became known as cascade theory, leading to the concept of the *ideal cascade*, which for a rate-based separation such as gaseous diffusion or thermal diffusion corresponds to both minimum stages and minimum energy consumption.

The rate-based separation processes which were developed for separations of heavy isotopes have the feature that they are inherently irreversible, being dependent upon differing rates of transport across a barrier without change of phase from feed to product. These I call *rate-based separation processes*, not to be confused with the “rate” processes of Foust, *et al*, which are something else. The minimum-stages and minimum-energy-consumption characteristics of the ideal cascade are specific to rate-based separation processes. Benedict (1947; Benedict &

Pigford, 1957) extended the concepts of cascade theory further to “potentially reversible” and “partially reversible” equilibration-based separations and to squared-off cascades, of the sort that are found in the more common process engineering separations such as distillation. The distinction between the ideal cascade and squared-off cascades is that the ideal cascade involves the generation of reflux at every stage, whereas a squared-off cascade operates without generation of reflux on internal stages. Generation of reflux at intermediate points is far simpler for rate-based processes than for equilibration separation processes, because of the lack of phase change and because persistence of reflux from stage to stage requires the addition of energy at each stage in rate-based processes but does not in equilibration processes.

The work of Cohen, Benedict and co-workers on isotope separation processes produced a number of useful concepts, among them

- theoretical interpretations and understanding of energy requirements and available-energy concepts for separations;
- the distinctions among potentially reversible, partially reversible, and irreversible separation processes; and
- the concepts of ideal distillation, uses of intermediate reflux and boil-up, and thermal linking of distillation columns to maximize the use of a given temperature span between heat sources and heat sinks.

The concept of thermal linking of distillation columns came as well from the development of distillation processes for the fractionation of air (Dodge, 1944). Potentially reversible, partially reversible and irreversible processes have now become more commonly known as equilibration processes with energy separating agents, equilibration processes with mass separating agents and rate-based separations, respectively.

ANALYTICAL CHEMISTRY, CHROMATOGRAPHY AND SEPARATION SCIENCE

Separations have always had their place in analytical chemistry, as any student of qualitative or quantitative analysis knows. Historically, the sorts of separations used in analytical chemistry have for the most part been different from those used in chemical processing. Reasons for this difference are the need for essentially complete separation in quantitative analysis and the fact that costs associated with consumption of reagents and formation of by-products are of little consequence in analytical chemistry but are of major importance in chemical processing. It is interesting to note that chemically driven precipitation is extensively used as a separation process in analytical chemistry, but is much less common in chemical processing.

An exception to this distinction is solvent extraction, which has found widespread use in both chemical processing and analytical chemistry. Solvent extraction, especially as applied to metallic and inorganic species and even as applied to many organic species, involves chemistry heavily. This fact has been recognized by the longstanding series of International Conferences on Solvent Extraction (ISECs), which include solvent extraction chemistry, solvent extraction in chemical processing and solvent extraction in analytical chemistry, all in an intimate mixture. In that solvent extraction of metallic species is usually based upon liquid ion exchange, there is also a close link between solvent extraction and ion exchange.

New dimensions in separations chemistry, analysis and processing arose with the development of gas and liquid chromatography in the '40s, '50s and '60s.. Chromatographic methods of separation afford the wherewithal of separating individual components within complex mixtures, a much needed aspect of analytical chemistry. Gas and liquid chromatography have become workhorse methods of chemical analysis. They have spawned a number of journals dedicated exclusively to chromatography, as well as a subdivision of the American Chemical Society's Division of Analytical Chemistry, known as the Subdivision of Chromatography and Separations Chemistry. Some of the advances in chromatography important to the general field of separation processes have been

- the development of complexing agents and methods of mounting complexing groups onto solid substrates,
- the development of the theory of chromatographic fractionation among solutes, and
- the development of methods of minimizing channeling and other methods of axial dispersion in fixed beds.

By its nature, classical elution chromatography has relatively low capacities for product throughput, and this has held back its use in chemical processing. However, we are now seeing much more widespread use of chromatographic techniques in process separations resulting from the ability of chromatographic methods to handle complex biological molecules without degradation, and from capacity-enhancing designs, such as those using progressing positions of multiple beds or the progressive movement of feed points in stationary beds.

The great attention given to chromatography has also led to the development of new methods of separation that are analogous to gas and liquid chromatography. Examples include gel-permeation chromatography, affinity chromatography and membrane chromatography. There also came into being a series of rate-based analogs to classical chromatography, starting with the invention of field-flow fractionation by Calvin Giddings. These developments led Giddings to start the journal *Separation Science* in 1966. This journal grew to include a variety of developments in separations chemistry and eventually changed its ti-

tle in 1978 to *Separation Science & Technology*, in which form it too provides a blend of separation chemistry, analytical methodologies and separations in chemical processing.

Another important general area of separations with roots in chemistry is membrane-based separations. Membrane-based separations received a boost with the development of the Loeb-Sourirajan asymmetric cellulose acetate membrane for purification of sea water and brackish waters, and with the development of macroporous membranes with relatively uniform pores. Incorporation of chemically active functional groups of the sort developed in extraction and chromatography has also been a useful feature.

From the chemistry viewpoint, there has become a field of separation science, noted already in the title of the journal by that name. Separation science connotes the applied chemistry and other fundamental knowledge underlying separations, including analytical separations. An elementary text edited by Karger, Snyder and Horvath (1973) surveyed that field. The Subdivision of Separation Science and Technology of the American Chemical Society's Division of Industrial & Engineering Chemistry was developed to cover separation science, as well as chemically based aspects of separation process technology.

Finally, the Gordon Research Conference on Separation and Purification was one of the first Gordon Research Conferences formed in the 1930s. It continues to this day, with the goal of serving both chemists and chemical engineers and working with underlying chemistry, chemical processing and analytical chemistry. A related Gordon Research Conference covers Reactive Polymers, Adsorption and Ion Exchange. The Gordon Research Conferences, the International Solvent Extraction Conferences, the Industrial and Engineering Chemistry Division of ACS and its journal *Industrial and Engineering Chemistry* (later *Industrial and Engineering Chemistry Research*), the journal *Separation Science and Technology* and the various developments surrounding chromatography have all played important roles in integrating chemical knowledge into the field of separation process technology.

SEPARATION PROCESSES

I will turn now to those developments that have served and facilitated the evolution from unit operations and mass-transfer operations to separation processes and separations technology as a field unto itself.

Interestingly, one important driver in this direction was the publication in 1960 of the book *Transport Phenomena*, by Bird, Stewart and Lightfoot. This landmark effort put the theories of fluid flow, heat transfer and mass transfer on powerful fundamental bases. Courses on transport phenomena appeared thereafter, and subsumed the aspects of fluid dynamics and heat and mass transfer that

had typically been included within courses on unit operations and mass-transfer operations.

Over the years, numerous texts and graduate courses had grown up, covering individual methods of separation. Examples the books by Robinson and Gilliland (1922, 1930, 1939, 1950), Hengstebeck (1961), and Van Winkle (1967) on distillation; by Treybal (1951, 1963) on extraction; by Sherwood and Pigford (1937, 1952) on absorption and extraction; by Mantell (1945, 1951) on adsorption; and by Helfferich (1962) on ion exchange.

The development of the field of transport phenomena coupled with a recognition that graduate-level courses on individual unit operations and methods of separation were relatively inefficient, in that there were many common features that could be covered in more efficient fashion in courses covering several, or even all, methods of separation.

In the 1960s several books sought to cover separation processes in a more comprehensive way. The growth toward a unified treatment of separations was incremental. Hanson, et al. (1962) and Holland (1963) pioneered treatment of digital computation methods for distillation and extraction. B. D. Smith (1963) treated the design and analysis of multistage columns for distillation, azeotropic and extractive distillation, absorption and extraction, concentrating upon binary separations. Oliver (1966) generalized phase equilibria, calculational methods and criteria for selection among processes and equipment, dealing with those separation processes commonly used in the petroleum industry.

Pratt (1967) authored the first book to consider the general theory of separation processes in a comprehensive way. Building on cascade theory and concepts of minimum energy of separation and loss of available energy from the Manhattan Project work of Cohen and Benedict, he contrasted equilibration and rate-based processes and treated several individual processes within each category, especially binary distillation, extensively. Although a milestone, Pratt's book achieved only limited use, resulting probably from the fact that the applications of cascade theory and related concepts were still rather well removed from practical applications for those separations of most general interest to chemical engineers.

During this same period of time, I was writing my own book, "Separation Processes" (King, 1971, 1980). I will give a bit of a digression to outline how this book came about. The project began upon my arrival at Berkeley as an Assistant Professor in 1963 when Don Hanson indicated that he was considering developing his extensive class notes on distillation into a book and asked if I would like to join him in the project. The totality of my relevant background was a doctoral dissertation on packed-column mass transfer, a two-year directorship of an M. I. T. Practice School station at an oil refinery, a jointly authored review chapter on absorption with chemical reaction, development of a case problem on multieffect evaporation, and having taught a graduate course in distillation once at M. I. T.

Nonetheless, in the naivety of youth I gratefully accepted and plunged in. Don and I met a few times to discuss the nature of the book and to divide up the drafting of the chapters. A necessary decision was whether it should be a book primarily on distillation or one on separations in general. In a snap judgement, based upon not much insight, I urged the general separations approach, and Don agreed. This discussion and decision took five minutes at most.

Within the next year I drafted a chapter and a half, and Don Hanson became Chair of the Berkeley Chemical Engineering Department. To my surprise one day, he indicated that he didn't see how he could make any progress on his chapters while being department chair, and therefore why didn't I just go ahead and write the book myself? With much of a summer invested in the chapter and a half, I decided that I had better proceed forward and protect my investment of time. The completion of the project took the better portion of each of the next five summers!

As I considered what was pertinent to separation processes in general, I endeavored to establish the functions and common features of separation processes, the reasons for staging and countercurrency, and the features and capabilities of countercurrent, cross-current, co-current, fixed-bed and elution chromatographic contacting. I then covered analytical, graphical and computer-based calculational methods for both binary and multicomponent single-stage separations and multi-stage separations, contrasting which features of different separation processes warranted which different calculational approaches. Following leads from Hengstebeck (1961), I pursued generalized use of the McCabe-Thiele diagram to illustrate patterns of compositional change within multi-stage separations, including multi-component distillation and non-distillative methods of separation. Working forward from Benedict (1947) and Pratt (1967), I included a chapter on energy requirements for separations, including concepts of reversibility, available energy consumption and the like. Finally I treated as relatively new subjects the logic of selecting types of separation processes for a given application, choosing sequences for multi-step separations, and optimizing designs. In the second edition (1980), I added treatment of basic mass-transfer concepts and eliminated the separate chapter on optimization.

Subsequently we have seen useful handbooks on separations technology edited by Schweitzer (1979) and by Rousseau (1987), as well as books by Henley & Seader (1981) and Humphrey and Keller (1997), the latter covering equipment, alternative processing methodologies and choices among approaches for distillation, extraction, adsorption and membrane-based separations.

Two other significant events in defining separation technology as a field were the launching of the Engineering Foundation Conferences on Separation Technology by Norman Li and associates in 1984 and the National Research Council study leading to the 1987 report, "Separation and Purification: Critical Needs and Opportunities" (King, et al., 1987), which defined, categorized and prioritized research needs in separations.

Finally, in 1991 the American Institute of Chemical Engineers made the significant change of creating a Separations Division and reorganizing and renaming Group II of the National Program Committee from Unit Operations to Separations, corresponding to the new division.

Although the emergence of a field of separation processes has been presented here as a sequence of rather isolated events and publications, it has also been enormously reinforced by the growing importance of a large number of different separations in industry. A greatly widening spectrum of industrial needs and applications has coupled with the development of newer methods of separation, such as membrane-based processes, such that many more different methods of separation are in common use. A related phenomenon has been the evolution of chemical engineering from a profession dealing very largely with the petroleum and chemical industries to a general-purpose profession centrally important in a wide variety of additional applications—pulp and paper, biological processing, environmental control, water purification and softening, pharmaceuticals, food and beverage processing, and production of personal and household products, among others.

WHAT IS GAINED BY A UNIFIED VIEW OF SEPARATIONS?

There are several major gains in understanding, insight, capability and efficiency that come from viewing separation processes as a unified field

1. The first such gain recognized historically is that methods of analyzing the degrees of separation achieved in different separation processes are similar and differ from one another in ways that are attributable to the characteristics of particular methods of separation. These methods of analysis include:
 - (a) calculations for single-stage, e. g., adiabatic flash, equilibrations;
 - (b) y - x or McCabe-Thiele analysis for binary, or binary-equivalent, separations;
 - (c) triangular and Janecke or Ponchon-Savarit diagrams for binary separations with a conserved quantity (a third component, enthalpy balance, etc.);
 - (d) group methods of calculation, e. g., the Kremser equation and like approaches;
 - (e) analytical methods for continuous-countercurrent, or differential, contactors, and
 - (f) digital computation methods.

Developments of powerful computational algorithms, immense gains in computing capacity following “Moore’s Law”, and the pervasive

and near universal spread of personal computers have meant that much less attention has to be given to methods of calculating degrees of separation, thereby allowing for much more emphasis on process selection, synthesis and improvement, both in university-level courses and in engineering practice.

2. The interactions of mass transfer and phase equilibria and their resultant effects are similar for related types of contacting equipment (e. g., perforated-plate and packed columns), even for different methods of separation. The concepts used for stage efficiencies are common, as well.
3. A unified view makes it possible to identify and select among candidate separation processes for a given task on a knowledgeable basis.
4. An understanding of solution and complexation chemistry makes it possible to identify and select among potential mass separating agents for different applications and to transfer the use of particular agents and chemical functionalities among different types of separation processes, e. g., solvent extraction, ion exchange, reversible chemical adsorption and absorption, chromatography and membrane-based processes.
5. Insight into the capabilities of a variety of methods of separation helps one identify when the ability to separate will pose a major process limit. One example is the separation of optical isomers, where there is major incentive for avoiding the separation altogether by appropriate choice of the method of synthesis.
6. A general understanding of separations facilitates generating entirely new methods of separation. Some examples from the past include the development of azeotropic and extractive distillation by Othmer and associates, the development of field-flow methods of separation by Giddings and associates, and the development of numerous variants on reverse osmosis and microfiltration.
7. A comprehensive knowledge of separations enables one to transfer the uses of separations to different scales of operation, ranging from analytical separations to large-process separations.
8. The logic and criteria for choosing effective sequences of individual separations are general.
9. The benefits and opportunities for increasing degrees of separation through countercurrency and staging apply across the different separation processes. As well, there are common reasons (intensification of contact without encountering flooding limits, no benefits of staging for enhancing the degree of separation) for when co-current or single-stage design is desirable or acceptable.
10. An understanding of patterns of stage-to-stage changes in composition in countercurrent separations and the causes for particular patterns is

useful across the board for improvement of design and operating conditions to gain a greater degree of separation and/or lesser flows and equipment cost.

11. An understanding of the factors governing energy consumption enables greater insight into reducing energy consumption and/or achieving an optimal combination of equipment and operating costs.
12. Finally, for university-level education, separations is one of the few aspects of the chemical engineering curriculum that are truly engineering, as opposed to applied science or engineering science. Along with reactor design and process design, separation processes afford excellent opportunities for teaching chemical engineering students to do engineering, *per se*.

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