A Course in

SEPARATIONS AND RECOVERY PROCESSES*

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T HERE ARE MANY INGENIOUS methods which can be used to "demix," or separate, feed streams into two or more streams of different composition. Historically, separation and recovery processes have played a major role in chemical engineering practice. Industrial methods for producing the required quantity of a particular product, of the desired quality, have often depended on the economics of the reactor as well as the separation and recovery processes. These latter costs, which in many cases dominate the overall costs, depend critically on the value of the particular substance and its concentration in the feed mixture.



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Until a few years ago, typical courses in separation processes usually covered the traditional processes of distillation, absorption and extraction. Many of these courses were based on C. Judson King's text, Separation Processes [1]. More recently, such courses have begun to introduce the relatively newer separation processes such as adsorption, chromatography, and ion-exchange [2], and membrane processes such as hyperfiltration and ultrafiltration [3]. The goals of the particular course described here are several: (1) to use the approaches developed in the study of transport phenomena [4, 5] as a unifying foundation; (2) to provide the student with an understanding of the design and operation of these processes; and (3) to review the particular advantages and limitations of the various processes being studied. Since graduate students with environmental (e.g.,water and wastewater treatment), chemical, and biochemical engineering interests attend this course, examples and applications from these areas are used.

PEDAGOGICAL APPROACH

Philosophy and Scope

Separation and recovery processes provide a fertile ground for applying the principles and tools which the students have learned in transport phenomena courses. In general, as is well-known, the transport of mass in many processes can be divided into several serial steps, each of which can be described by a characteristic velocity and length. Often the mass transport occurs simultaneously with momentum and/or energy transport, complicating the analysis. In addition to the differences in the dynamical aspects of these processes, they can also be differentiated by the extent to which equilibrium is attained. The separation processes covered in this course are classified in these ways; this classification is presented in

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Table 1, in which mechanical (differential density and size), equilibrium (sorption), and rategoverned (differential pressure) processes are shown. One of the primary reasons for choosing these three types of processes is that they are among the major separation operations used in water and wastewater treatment. Moreover, they are currently being considered and, in some cases, already being used for separations in the newlyevolving biotechnology industry. (This is due, in part, to the fact that many of these processes operate under isothermal conditions without requiring a phase-change for separation, an attractive advantage in many instances.)

Lectures and In-Class Discussion

The structure of the course is outlined in Table 2. Initially, some basic fundamental definitions and necessary concepts are reviewed. Following this

brief review, the lectures are organized into three main categories: differential migration, filtration, and sorption. With the exception of the course project, the subject matter is presented in the order indicated in Table 2. With respect to the project, however, the status report and the final presentation are given at the midpoint and at the end of the schedule, respectively.

The various indices which are used to measure performance and to compare different separation processes are defined at the outset of the course. Separation factors and fluxes are evaluated in terms of the operating variables in order to identify the dominant process variables with respect to separation efficiency and rate. In many processes, reaction continues after the process stream leaves the reactor and enters the separator; in addition, solute dispersion in the separator often can directly effect the separator efficiency.

Туре	Driving Force	Example	Covered
Mechanical (heterogeneous feed)	a. Density difference	Settling, centrifugation, flotation	yes
	b. Size difference	Filtration, hydrodynamic, chromatography, membrane	yes
	c. Surface (interfacial)	Flotation, gel filtration	no
	d. Magnetic gradient	High gradient magnetic filtration	no
	e. Inertial gradient (hydrodynamic)	Membrane, filtration	yes
Diffusional (equilibrium,	a. Energy separatory agent	Distillation, drying	no
homogeneous feed)	b. Mass separatory agent —Gas	Absorption	no
	—Solid, liquid	Sorption (absorption, ion exchange, affinity)	yes
		Stripping, extraction, precipitation	no
	c. Gradient equilibration	Isoelectric focusing	no
Rate governed (non-equilibration, homo/heterogenous)	a. Pressure gradient	Gaseous diffusion membrane filtration	yes
	b. Osmotic gradient	Dialysis	no
	c. Electrical field gradient	Electrodialysis, electrophoresis, electrolysis	no
0	d. Magnetic field gradient	Mass spectroscopy	no
	e. Chemical potential gradient	Facilitated transport	no

TABLE 1 Classification of Separation Processes [1]

Hence, a review of selected concepts of mixing in reactors and reactor models in (separator) vessels is presented [6]. In particular, this is relevant and useful to environmental engineering students who may have a weak background in chemical reaction engineering.

Since the separation of multiphase or heterogeneous feeds is considered in this course, it is important to review the fundamental bases of colloidal destabilization [7-10] and of flocculation [11, 12]. A critical factor influencing flocculation is the effect of mechanically or hydrodynamicallyinduced shear; hence, this topic is also briefly considered [13, 14].

As an introduction to the material on differential migration processes, some simple analysis

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TABLE 2

Course Outline: Separations and Recovery Processes

- 1. Review (2 weeks)
 - Definitions for flux and separation factor
 - Models for ideal and non-ideal flow in vessels [6]
 - Destabilization and transport of colloids [7-12]
 - Mixing [13, 14]
- 2. Differential Migration (2 weeks)
 - Motion of particles in a fluid [15]
 - Sedimentation [16]
 - Centrifugation [14, 15]
 - Flotation [17]
- 3. Filtration (3 weeks)
 - Flow through porous media [15]
 - Deep bed [18-24]
 - Cake [20]
 - Hyperfiltration, ultrifiltration and microfiltration [26]
 - Equipment [25]
- 4. Sorption (4 weeks)
 - Types-adsorption, ion exchange, and affinity
 - Nature of sorbents [33-35]
 - Equilibrium processes—gas/liquid [44-46]
 - Rate processes (kinetics)—batch, fixed and expanded bed [45, 46]
 - Modes of operation [45]
- 5. Selection of a Separation Process [1] (1 week)
 - Energy requirements
 - Factors for process selection
- 6. Project $(1 \ 1/2 \ weeks)$
 - Interim status report (10 minutes)
 - Final presentation (40 minutes)

of the motion of particles in a fluid is presented [15]. For example, equating the Stokes drag force for a sphere $(3\pi\mu du)$ with the buoyant force, the terminal velocity for each process is derived. Following this basic general discussion, each of the various processes is considered in detail. Some of the topics covered include, for example, development and comparison of design equations for bowl and tubular centrifuges, and analysis of velocity theory for types 1-4 settling. Also, sludge withdrawal theory is presented, using the solids flux versus concentration approach [17]. Finally, mass balances are made around various flotation systems (with and without recycle) in order to obtain expressions for the air to solids ratio [17].

Various types of filtration processes, all of which involve the movement of fluid through a porous media (e.g., flow past the grains in a depth filter or flow through a synthetic septa or membrane) are presented in this class. Hence, the Carman-Kozeny equation is derived from fundamental principals [15] in this course. Further, the analogy with pipe flow is extended and Carman's results for modified friction factors versus modified Reynolds numbers are presented for laminar and turbulent flow conditions. The mechanisms important to filtration are presented; e.g., the classical capture mechanisms including capture resulting from Brownian diffusion, interception, inertial impaction and lift, and gravitational deposition are presented and used in a dimensional analysis with Iwasaki's filtration equation. The concepts and use of particle trajectory calculations are introduced using Spielman's [18] and Rajagopalan and Tien's [19] analyses. Application of these principles to water and wastewater filtration is then considered [20-22] in order to illustrate the concepts, etc., discussed. Transient behavior in deep bed filters and the effects of polymers on particle adhesion mechanisms are discussed next [23, 24]. The effects of precoats and body-feeds (e.g., diatomaceous earth) and how their concentration affects dynamic pressure drops and other variables are discussed. Lastly, selected examples of typical flow sheets for deep bed and cake filtration are discussed [25].

Membrane processes are covered in another graduate course, "Advanced Membrane Concepts"; hence these are not discussed in detail in this separations course [26]. Instead, they are mentioned briefly, and general concepts and overview are given here. Membrane processes can be characterized by their driving forces (pressure,

CHEMICAL ENGINEERING EDUCATION

concentration, and electrical gradient) or by the species removed from the feed stream (solvent, neutral, or charged solutes). Examples of pressure-driven membranes processes include hyperfiltration (also called reverse osmosis), ultrafiltration, and microfiltration. One example of a concentration-driven process is dialysis, and one example of an electrically-driven process is electrodialysis. However, only the pressure-driven processes are covered here.

The major advantages of these pressure-driven membrane processes, e.g., that they operate without a phase-change at relatively constant temperature and that they are relatively less expensive to operate (for such applications as seawater desalination) when compared to other processes such as distillation [27], are emphasized. In addition, a summary of membrane formation from different commercial materials is presented. The physicochemical bases of membrane formation and morphological structure are then discussed using ternary diagrams to distinguish between hyperfiltration and ultrafiltration membranes [28]. A general formalism for membrane transport of solvent and solute is presented using the framework of irreversible thermodynamics, and well-known transport models such as the Spiegler-Kedem model, the solution-diffusion model, and the finely-porous model are compared [29]. Other concepts developed are those of concentration polarization and membrane fouling, where osmotic pressure or gellimitation of the wall solute concentration is compared [30]. Correlations for mass transfer of proteins and cells to the membrane-solution interface are analyzed and selected models are contrasted and compared. For example, the discussion here emphasizes that the gel-polarization model of Michaels [31] is most appropriate for macromolecular solutions while the gel-polarizationlateral-migration model of Belfort, et al [26] is useful for colloidal suspensions and mixed feeds which contain macromolecules or colloids. The final discussions on membranes focus on different membrane module designs; these are compared on the basis of such variables as permeation area/ volume ratio, cross-flow fluid mechanics, and scaleup potential. The importance of fluid mechanics and momentum and mass transfer are illustrated in Fig. 1 and show the various velocity and concentration profiles for cross-flow filtration.

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FALL 1985

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FIGURE 1. Mass transfer in porous ducts with suction.

tion to this portion of the course is a review of a range of applications. After this, the dominant forces and interactions between solute and sorbent are discussed. The roles of these in different processes are described and contrasted; for example, it is noted that the London dispersion forces are important during the adsorption of organics from aqueous solution onto microporous activated carbon [32], electrostatic interactions are important in ion-exchange, and hydrophobic interactions may be significant in certain bio-affinity sorption processes. Properties used to characterize the nature of sorbents, e.g., mechanical and sorptive properties such as specificity (selectivity) and capacity, are discussed. Some of the relevant material presented includes a brief overview of preparation, properties, and applications of sorbents used in adsorption, ion-exchange, and affinity processes [33-35].

The mass transfer principles (e.g., solute transport and attachment) needed in analysis of these processes are discussed first, qualitatively. Following this, the relevant quantitative principles, etc., are given. For example, the Gibbs-Duhem equation (for equilibrium cases) and the Gibbs adsorption isotherm (for constant temperature cases) are derived [36, 37]. Subsequently, theoretical expressions for two-dimensional surfaces are derived (for systems satisfying certain diluteness' assumptions) in order to obtain an equation for the spreading pressure [38]. This leads easily to the next topic, which is a discussion of experimental methods to obtain the spreading pressure as a function of coverage. Concepts relevant to competitive adsorption, using both ideal Continued on page 215.

SEPARATION PROCESSES

Continued from page 175.

adsorbed solution theory [39, 40] and non-ideal adsorbed solution theory [41], are described. The thermodynamics of adsorption are presented based on the 'surface excess' approach, and applications (for example, formulation of a consistency criterion for the adsorption of three mutuallymiscible liquid pairs, e.g., adsorption of benzene, cyclohexane and n-hexane on graphitized carbon [42, 43]) are covered in detail. A comprehensive thermodynamic formalism is then developed, using the solvophobic approach for the association reaction between solute and sorbent in the presence of the solvent. Applications of the results of this are then used to correlate the change in calculated free energy of adsorption (which depends on chemical structure) with amount adsorbed [32, 44]. The utility of this approach, e.g., determination of which one of a benign or toxic isomer would preferentially sorb during activated carbon adsorption is illustrated. Students are shown several useful isotherm models, including the Langmuir, BET and Freundlich expressions. Finally, the kinetics and rate processes of fixed bed sorption are presented in detail [45, 46]. Diffusion and sorption phenomena inside a porous sorbent are used to explain the sorption kinetics in a homogeneous granule (*e.g.*, as a function of effectiveness factor) [4]. Break-through curves and solutions to mass transfer problems in sorption columns using orthogonal collocation methods are described and compared [47]. This portion of class discussion concludes with a short description of chromatographic separations [48, 49].

The final subjects covered in this course are the energy requirements for separation and the factors significant in process selection. The student is referred to Chapters 13 and 14 of King's text [1] for an overview of these topics.

Assigned Readings

Approximately every two weeks, a paper from the engineering or science literature, which is relevant to the subject matter under discussion in class, is assigned. The students are given one week to study the paper; then, an in-class session (approximately 15 to 30 minutes) is devoted to discussion, critique, and evaluation of the assigned paper. Review papers and specific research papers are chosen to supplement the formal lecture in this manner. Students are expected to lead the discussion—however, guidance and encouragement

TABLE 3

Suggested Topics for Project (Term Paper)

- 1. Affinity separation methods: scale-up and new developments.
- 2. Cross-flow microfiltration: new developments for cell harvesting.
- 3. Removal of pathogens for potable water.
- 4. Dispersion analysis: uses in separation and recovery processes.
- 5. Membrane processes in municipal water treatment: review of problems.
- 6. Gel-filtration: scale-up potential.
- 7. Organics separation by reverse osmosis.
- 8. Lamellar settlers: recent developments.
- 9. Treatment of the leachate from the Love Canal (or other chemical waste dumps).
- 10. Breakthrough dynamics in sorption systems: models and limitations.
- 11. Fluidized carriers for separations.
- 12. Disruption of cells: a comparison of different methods.
- 13. Comparison between centrifugation and cross-flow filtration for downstream processing of bio-reactors (fermentors).
- 14. Recent developments in large scale fractionation by chromatography.
- 15. Recent developments in immobilized reactors.
- 16. Design an interactive graphics instructional unit relevant to course.
- 17. Student's suggestion, with instructor's approval.

from the instructor is often needed. One purpose of these critical review sessions is to expose the student to the current engineering and science literature, especially to those journals relevant to the separation field.

Report and Homework Assignments

A term paper or report (maximum length of thirty typed pages) is required from each student at the end of the course. The students may choose any topic (in consultation with the instructor) related to separation and recovery processes (see Table 3 for a sample list of suggested topics). Four steps are involved in preparing this report. The first step is the selection and approval of the subject of the report. Secondly, the student prepares a title and one-page outline of the proposed report; this is given to the instructor three weeks after classes begin. (The time scale permits the instructor to suggest changes, if necessary.) Third, the following instructions are then given to the student: "The student should (1) use the current literature to review and analyze the chosen topic. in order to present a state-of-the-art overview of the subject; (2) not reproduce ideas of others verbatim unless placed in quotes; (3) try to give his/her own views on the subject; and (4) the term paper should be written in the format acceptable for publication for a (specified) journal." Approximately mid-way through the course, each student is expected to present a 10-minute interim presentation reviewing the status and future plans for his/her report. Fourth, the student is graded on the term paper (15% of the final course grade), the 10-minute interim presentation (3%), and the formal 20-minute final presentation (7%). The term paper is due before the student gives the final presentation, near the end of the course. The final presentation is graded with respect to both the quality of the presentation itself as well as that of the content.

Homework assignments, which consist of several problems per assignment, are assigned every two weeks (these are one-week out-of-phase with the literature critique sessions). For each assignment, one or two students are each asked to set a problem and to provide its solution. Twentyfive percent of the final grade is determined by performances on homework assignments and literature critiques in class.

Exams

Two exams (worth a total of 50% of the final grade) are given. One exam covers the first half of the course (usually this corresponds to material up to and including cake filtration) while the second exam covers the subject matter from the second half of the course only [see Table 2]. The exams are either open- or closed-book for a duration of about two hours. Occasionally a take-home exam has also been assigned.

Text and Readings

Currently, there is no suitable text which includes all the subject matter covered in this course; hence, an assortment of several recent texts and review papers, each of which covers material relevant to different referenced parts of the course, is used. These texts and papers are referenced in Table 2.

CONCLUSIONS

Many new and cost-effective separation and recovery processes have been developed during the past twenty years. Current and future students must learn about these processes in order to understand the fundamental nature of these (and other) processes, the associated limitations and advantages, and operation and design con-

siderations. In the course described herein, the author has developed a unifying approach to me-(differential density), equilibration chanical (sorption), and rate-governed (differential pressure) processes. Traditional lecture format and in-class discussions of assigned papers from the separation literature are used in this course. Other components of the course include required biweekly problem sets and regular readings to supplement the lecture notes. One major component of the course is the report (this includes the associated interim and final report presentations). One innovation introduced in this course is the requirement that students prepare (with guidance if necessary) a problem, complete with its solution, for the biweekly homework sets.

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DEDICATION

This paper is dedicated to the late David Hansen who during his tenure as Chairman encouraged intellectual exploration and allowed the author to develop the course described here.

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