AC 2012-3671: AN INSTRUCTIONAL MODULE ON HYBRID SEPARATIONS FOR UNDERGRADUATE CHEMICAL ENGINEERING SEPARATIONS COURSES

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Instructional Module on Hybrid Separations for Undergraduate Chemical Engineering Separations Courses

Abstract

In industry, chemical engineers are often enlisted to retrofit equipment and/or modify operations to achieve increased production capacity, increased product purity and/or improved energy efficiency. In undergraduate chemical engineering separations courses, the focus is on stand-alone separation processes such as distillation, gas absorption/stripping, and extraction. At some universities, membrane-based processes are included in the traditional separations course. It may often be advantageous to couple two separation processes to achieve a superior separation sequence in terms of operating costs and/or capital expenditures. Separation of an azeotropic mixture using distillation alone requires the introduction of an extractive solvent or an entrainer, either of which much be recovered and recycled. The benefits of hybridization in separations are realized for the separation of azeotropic mixtures using distillation and pervaporation. An instructional module on hybrid separations was developed for incorporation into the undergraduate separations course.

Introduction

The subjects of separations and chemical reactor design are unique to the chemical engineering curriculum. These subject areas offer many opportunities for innovation using process intensification concepts including combining separation and reaction in a single process unit to overcome equilibrium limitations associated with reaction. It may also be possible to significantly reduce energy consumption associated with chemical production routes through combining two separation processes, each well suited for a particular composition range. Recent progress in hybrid separations is manifested in the growth of journal publications, government reports and patents that have been produced during the past decade. A variety of different separation processes have been coupled in hybrid systems including pressure swing adsorption and simulated moving beds [1,2], pressure swing adsorption and gas permeation [3], chromatography and crystallization [4], distillation coupled with vapor permeation [5-9], distillation with gas permeation [10], and distillation with pervaporation [11-14]. Additionally, a number of review articles have been published discussing the needs with respect to research in the area of process intensification to address sustainability in chemical processing [15-19].

In traditional undergraduate curriculums, separation processes are often taught following the 'unit operations' approach; each process is analyzed/discussed for performing the entire required separation of a given mixture. However, the advantages of coupling two distinct separation processes, each of which may offer benefits with respect to separation in different composition ranges, is often overlooked in the undergraduate curriculum. An instructional module on hybrid separation processes has been developed for the separations course that provides information regarding the advantages/disadvantages of conventional separation processes (distillation, extraction) as well as for some of the more recently developed separation processes (pressure swing adsorption, pervaporation, gas permeation). Within the module, case studies are used to demonstrate the superior performance of hybrid systems coupling these conventional and more recently developed separation processes.

Overview of Separations Course at Mississippi State University

The junior level separations course is taught on a traditional semester. The text used is Separation Process Engineering: Includes Mass Transfer Analysis [20]. Topics included in the course are shown in Table 1.

This course is taught on a 14.5 week semester, so covering the given topics is a challenge. However, by midsemester, the first seven topics have been covered in depth. This allows a comprehensive design project to be assigned where a distillation process for a given multicomponent mixture is designed to meet certain processing constraints (production rate, product purity). A commercial process simulator (Chemcad) is available for students to use in the project. In this open-ended project, the student teams also must size and cost the columns, reboilers, and condensers, as well as determine utility usage and operating costs. The given mixture and processing constraints are always selected so as to require multiple columns to accomplish the specified separations. Additionally, it is typically not possible with the given constraints to simply adjust the process variables for one column without significantly affecting the operation of adjacent columns. Thus, this activity provides valuable experience in how column operating conditions influence downstream processing units. The design project is assigned at the halfway point in the semester with a due date approximately 1 week before the end of the semester.

During review, it was suggested that the design project be structured to compare a hybrid process with a conventional process for a given separation. While this activity would be very beneficial in terms of enhancing student exposure to hybrid separations, the design project, as structured, is the first 'open-ended' design project that these students have experienced. As a first experience, the focus of the project must necessarily be very narrow. When one of the co-authors (RT) has taught the Process Design course taken the semester immediately after the Mass Transfer Operations course, the first design project in that course is normally a comparison of two design alternatives, typically involving only separations. A subsequent design project examined the use of gas permeation for hydrogen recovery in an existing process for economic savings. Thus, having a hybrid separation as one of the alternatives or an addition of a membrane separation to an existing process could readily be incorporated into the assignments for the Process Design course.

Approximately 3 weeks are spent on absorption/stripping and liquid-liquid extraction. Approximations for dilute solutions are presented. While one or two examples where single solutes are present in the mixture are given, the majority of time on absorption/stripping is spent examining mixtures containing multiple solutes. The remaining 4 weeks are split equally between diffusional mass transfer concepts and membrane separation processes.

Motivation for Module Development

One of the challenges in this course is providing adequate coverage of topics that are pertinent to the industrial practice of separations, while providing sufficient exposure to the fundamentals of separations. For example, graduates from our program are much more likely to encounter multi-component distillation, azeotropic and extractive distillation, gas absorption and stripping and liquid-liquid extraction compared to either binary distillation, leaching, adsorption, or chromatography. Yet, sufficient exposure to binary distillation is necessary to provide the foundation for examining multi-component separations. The course must also provide a sound basis for engineering decision-making with respect to sequencing of separations, including the coverage of heuristics used in industry. The course must provide students with sufficient information regarding the changing trends in industry as well as with sufficient knowledge to adequately compare traditional separations processing strategies with novel technologies or approaches. During the curriculum development project, three stand-alone modules have been developed for the junior level separations course that focus on these novel technologies: 1) reactive distillation; 2) divided wall columns; and 3) hybrid separations. The coupling of two distinct separation processes to exploit the superior performance of each in select composition regions is the basis of hybrid separations. This paper focuses on the instructional module developed for hybrid separations. The other two modules are discussed in [21].

Table 1. Course Elements for Junior Level Separations Course

1. Phase equilibrium (VLE, LLE, VLLE)	10. Reactive distillation
2. Flash distillation, including drum sizing	11. Divided wall columns
3. Binary distillation (Tray-to-tray analysis, McCabe Thiele analysis)	11. Gas absorption and stripping including design for multiple solutes
4. Column configuration - operating modes for condensers, reboilers, use of steam, vacuum, elevated pressure	12. Liquid liquid extraction
5. Multicomponent distillation - composition profiles, sequencing of columns, heuristics for separations	13. Mass transfer coefficients
6. Shortcut method (Fenske-Underwood-Gilliland method)	14. HTU/NTU and HOG/NOG analysis
7. Use of simulation software for shortcut methods and for rigorous methods - Chemcad	15. Membrane separations (pervaporation, gas permeation)
8. Column design - trayed columns and packed columns	16. Other topics as time allows
9. Basic economics of distillation processes (column costing, determination of operating costs)	

Key Elements and Structure of Instructional Module

The instructional module is comprised of distinct sections, each designed to provide information regarding a key aspect of hybrid separation technology. A few of the very successful applications of hybrid separation technology include separation processes that are not currently discussed in depth in our junior level separations course. Thus, one section is an overview of separation processes that might be coupled in a hybrid system, with each overview providing information regarding basic operation of the process, range of compositions where the process possesses superior performance as well as its advantages/disadvantages.

It is assumed that the module will be used in a stand-alone format; thus, the module contains all pertinent background, instructional content, and applicable references. The sections of the module include:

- Introduction to Hybrid Separations
- Overview of Separation Processes
    - Distillation
    - Adsorption/Pressure Swing Adsorption
    - Gas Permeation and Vapor Permeation
    - Pervaporation
- Benefits Derived from Coupling of Separation Processes
- Assessment of Success of Hybrid Separation Processes
- Heuristics for Hybrid Separations
- Energy Intensive Separations - Potential Hybrid Successes
- Case Studies - Examples
    - Pressure Swing Adsorption/Membrane Permeation Hybrid
    - Distillation/Vapor Permeation Hybrid Process
    - Distillation/Pervaporation Hybrid Process
- Summary

Case studies will include comparison of the conventional technology (current state of the art as practiced in industry) and the hybrid technology, including capital costs and operating costs. Select screen shots of module content are shown for the case studies.

Case Studies - Examples

Case studies can be used to demonstrate the particular benefits achieved through coupling of separation processes. Three case studies are included in the initial version of the module.

Pressure Swing Adsorption/Membrane Permeation Hybrid

In dealkylation processes, a gas stream containing hydrogen and low molecular weight hydrocarbons (such as methane) is generated; the hydrogen present is reactant not consumed during the dealkylation process, while the methane is typically a product of the reaction. Recovery of the hydrogen for recycle can lead to potential energy savings. Membrane processes alone are typically not capable of producing high purity products [20].

Coupling pressure swing adsorption with gas permeation offers many different configurations for the hybrid process. The optimal configuration is influenced by a number of factors including which species is most permeable through the membrane, which species is most strongly adsorbed on the adsorbent bed, and operating temperatures/pressures [3]. In the case studies presented, different configurations along with their advantages/disadvantages and performance will be examined. Separation of hydrogen from methane (as realized in a petrochemical facility) as well as separation of nitrogen from methane (encountered in the upgrading of natural gas) are the two separations that are discussed.

Distillation/Vapor Permeation Hybrid Process

In this application, distillation is combined with vapor permeation to achieve the desired product distribution [8]. Two of the most energy intensive separation processes used widely in the chemical industry are the separation of ethane/ethylene and propane/propylene. Both chemical systems possess a relative volatility between components that is close to unity; thus, use of a single distillation process to achieve high purity product streams requires both high reflux and a large number of stages. In fact, in many installations, these large number of stages are split between two coupled columns, one acting as a stripper, the other as an enricher, in order to keep column height of a single column under approximately 150 feet. Due to the low boiling points of these species, cryogenic distillation is employed. A variety of configurations can be envisioned for the hybrid process [8], where the membrane unit is used primarily for: 1) distillate product finishing; 2) feed stream pretreatment; 3) treatment of a side stream with permeate/retentate being returned as a) an overhead product/reflux stream (permeate) and a returning side stream (retentate) entering the stripping section and b) a returning side stream (permeate) entering the enriching section and a second returning side stream (retentate) entering the stripping section. Optimal results are achieved using a membrane unit that operates under mode 3(b), resulting in minimum required membrane area and minimum condenser duty [8].

Distillation/Pervaporation Hybrid Process

One of the early commercial successes of pervaporation is the industrial dehydration of isopropanol (2-propanol). The presence of the azeotrope in this system requires the use of a mass-separating agent in extractive distillation, or an alternate operating mode, such as pressure swing distillation, where the sensitivity of the azeotrope to system pressure is exploited to overcome thermodynamic limitations associated with the presence of an azeotrope. The use of pervaporation coupled with distillation allows the use of distillation for bulk fractionation where its performance is most robust, and the exploitation of membrane permeability for removal of sufficient water by pervaporation to surpass the azeotropic composition. Wankat [20] discusses in detail the use of a hybrid distillation/pervaporation system for the separation of ethanol and water.

The focus of the module case study is the dehydration of 2-propanol. Sommer and Melin discuss this separation process and present a variety of possible configurations for the hybrid system [9]. The base case against which comparisons are made is the extractive distillation of 2-propanol/water in the presence of ethylene glycol (the extractive agent). Two hybrid configurations, one where a pervaporation unit is place between two distillation columns, and one where the pervaporation unit is placed in the reflux loop of a single distillation column, are considered [9]. The second configuration is identified as possessing the greatest reductions in capital and operating costs (40 % reduction) and energy consumption (85 % reduction) [9]. The commercially available membrane employed is a PVA/PAN polymeric membrane, which is hydrophilic. The concept of selecting a membrane such that the minor component in a stream is the component that preferentially passes through the membrane is presented in the instructional module. This case study, combined with the hybrid system examined in the text, demonstrate the potential for a hybrid distillation/pervaporation system for azeotropic systems.

Implementation

The module is designed as a stand-alone entity, currently as a Powerpoint presentation. During summer 2012, the module will be transferred to the Authorware platform for ease in dissemination. This module will be used in the separations course for the first time during the Spring 2012 semester. A pre and post-lecture questionnaire will be completed by the students to assess their understanding of important concepts in hybrid separations as well as its advantages and limitations. Assessment data will be available for the presentation, but are not available at this time.

Conclusion

A module is in development that will provide instruction on hybrid separation processes. This modules will include material to allow students to learn of the benefits derived from the coupling of two distinct separation processes. Exposure to the concept of hybrid separations as part of the undergraduate chemical engineering curriculum will provide graduates who have knowledge of hybrid systems and how this coupling can be advantageously used to reduce energy consumption and overall improve process energy efficiency.

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