

Understanding Batch Distillation Process Principles with MultiBatchDS

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ABSTRACT

This article describes the MultiBatch Distillation Systems (MultiBatchDS) for simulation, optimal design, and control of batch distillation columns with multiple configurations, multiple operating modes, and multiple fractions with multiple levels of models. The use of this package as a teaching aid for explaining batch distillation fundamentals is presented.

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INTRODUCTION

The recent increase in the production of high value-added, low-volume specialty chemicals and biochemicals has generated a renewed interest in batch processing technologies. Batch distillation is an important unit operation in the batch processing industry and is widely used. The flexibility of batch distillation, combined with the inherent unsteady nature of the process, poses challenging design and operation problems. In view of the practical importance of batch processing and in response to industrial demands for chemical engineers with a strong background in batch processing, more and more educational institutions are redesigning their curricula to include courses devoted to the subject.

A dedicated course on batch distillation will: (1) introduce the various configurations of conventional and emerging batch distillation columns; (2) exam-

ine the challenges involved in a rigorous modeling of batch distillation column dynamics; (3) describe the various operating modes in detail; (4) provide hierarchy of models of varying complexity and rigor; (5) present approaches to optimal design of batch distillation and highlight the differences vis-à-vis continuous distillation columns; (6) describe optimal control algorithms and its application to batch distillation; (7) discuss analysis and synthesis of columns with complex thermodynamics, and complex, unconventional column configurations; and (8) illustrate real-world applications.

It is difficult to teach batch distillation without using computers, because of the two reasons stated above: (1) the process is time varying and one has to resort to complex numeric integration techniques and different models for obtaining the transients, and (2) this ever-changing process also provides flexibility in operating and configuring a column in numerous ways. This article describes MultiBatchDS (BPRC Inc., 8001 Lingay Drive, Allison Park, PA 15101, email:bprc@bprc.com), a general-purpose batch distillation package that can

be used for understanding batch distillation process principles.

A description of the first version of this package named BATCH-DIST appeared in Diwekar and Madhavan [1]. However, MultiBatchDS has a large number of additional features with a Windows interface and it is available in educational and commercial versions.

Select features of MultiBatchDS include:

- a user-friendly graphic, input-output interface
- on-line help screens
- on-line monitoring and control of the program
- a data bank and thermodynamic models for predicting properties and vapor-liquid equilibria of ideal and nonideal mixtures
- capabilities for analyzing and optimizing complex and unconventional column configurations, including emerging designs, such as columns with a middle vessel and stripper
- dynamic simulation models with varying degrees of complexity and rigor
- rigorous models for detailed column design, based on solution of fundamental equations governing transient heat and mass transfer column operation
- simulation of start-up conditions
- simplified, yet effective models for preliminary design, synthesis, optimization, and rapid analysis of column behavior
- tools for optimization and optimal control of columns.

MODELING BATCH DISTILLATION COLUMN CONFIGURATIONS

The transient nature of batch distillation allows for configuring the column in a number of different ways, some of which are shown in Figure 1 [2]. The column in Figure 1a is a normal batch distillation column with a reboiler at the bottom and a condenser at the top, which essentially performs the rectifying operation. One column can be used to separate several products using the multifraction operation of batch distillation presented in Figure 1b. In the multifraction operation several cuts or fractions are made in a single column. Some cuts can be desired and others can be intermediate products. These intermediate fractions can be recycled. Figure 1c shows this periodic operation in which each charge consists of fresh feedstock mixed with recy-

clered off-specification material from the previous charge. Figure 1d represents the stripping column for separating light ends, where the storage vessel is kept at the top of the column in which the original mixture is fed. In 1994, Devidyan et al. [3] presented a batch distillation column that has both stripping and rectifying sections embedded in it (Fig. 1e). Although this column has not been investigated completely, preliminary analysis has demonstrated that it provides added flexibility for batch distillation operation. Recently Skogstestad et al. [4] described a new column called a multivessel column (Fig. 2) and showed that the column can obtain purer products at the end of a total reflux operation. These are a few examples of the different kinds of batch distillation columns. MultiBatchDS includes models of the different possible configurations as shown in Figure 1. The multivessel column model (Fig. 2) is currently being incorporated in the package.

START-UP CONDITIONS

The batch distillation startup period consists of the following three steps: (1) preheating the still charge to its bubble point; (2) filling the column and the condenser holdups; (3) running the column without distillate withdrawal and bringing the unit to a steady state [5].

At the end of the total reflux condition (step 3 above), the given charge distributes itself throughout the column. This effect is known as the capacitance effect. Associated with the capacitance effect is the concept of equilibration time, T_{eq} . The equilibration time is the time required from the time the column is charged to attain the steady state.

The second step can be achieved in different ways as given below:

- Charge the feed from the top so that initially the pot composition, as well as the composition on each plate, is equal to the feed composition.
- Charge the feed to the still and operate the column without reflux (zero reflux). This results in the composition on each plate being equal to the vapor composition, which is in equilibrium with the feed composition.

This mode was suggested by Luyben [6] but was never before applied in simulation or in practice [5]. The default start-up mode in the MultiBatchDS is the first such mode.

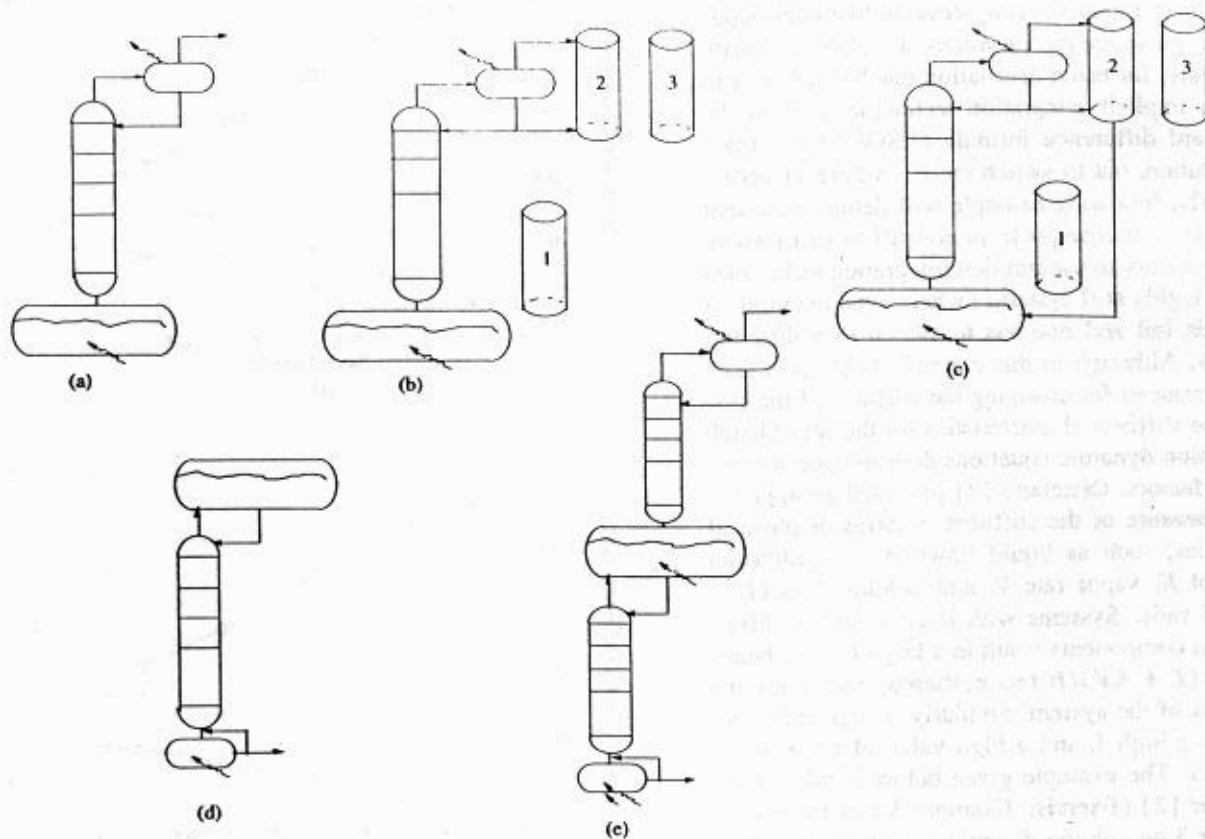


Figure 1 Examples of ways to configure the batch distillation column.

While simulating the complete dynamics of the start-up operation can be computationally intensive, one can get estimates of equilibration time, which

cannot be obtained from the steady-state model. Furthermore, many a time in the plant, the total reflux operation is interrupted before the equilibration time is reached. In this situation, one has to resort to the complete dynamic simulation of the start-up condition. Therefore, MultiBatchDS gives the option of simulating the start-up conditions using the transient or the steady-state model.

SOLUTION TECHNIQUE AND MODEL SWITCHING

A rigorous model of the batch distillation operation involves solving several stiff differential equations. The first detailed analysis of batch rectification dynamics was presented by Distefano [7,8]. He pointed out that because of several factors the system of equations presented for batch rectification is much more difficult to solve than that for the dynamics of a continuous column. For example, in the case of batch distillation plate holdups are generally much smaller than the reboiler hold-up, while in continuous distillation the ratio of reboiler to plate

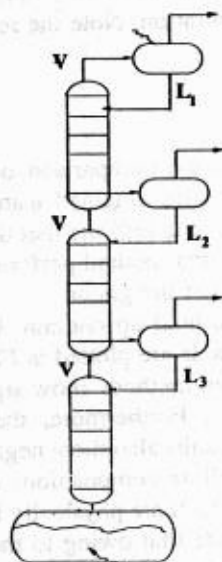


Figure 2 The multivessel column.

hold-up is not nearly as great. Also, in batch distillation severe transients can occur, unlike continuous distillation where the variations are relatively small. Therefore, for batch distillation one has not only to use an implicit integration technique such as the backward difference formula (LSODE) to obtain the solution, but to switch models wherever necessary. The following example will demonstrate that how a system changes from nonstiff to stiff (where it is necessary to use implicit integration technique) and to highly stiff systems (where even the implicit methods fail and one has to switch to a different model). Although in this example hold-up is used as a parameter for changing the stiffness of the system, the stiffness characteristics for the set of batch distillation dynamic equations depend upon a number of factors. Distefano [7] provided an approximate measure of the stiffness in terms of physical quantities, such as liquid flowrate L , equilibrium constant K , vapor rate V , and holdup H as $(L + KV)/H$ ratio. Systems with large volatility differences in components result in a large K , and, hence a large $(L + KV)/H$ factor, thereby increasing the stiffness of the system. Similarly, a high reflux results in a high L and a high value of $(L + KV)/H$ factor. The example given below is taken from Diwekar [2] (Exercise Example 3.1 at the end of Chapter 3 on column dynamics) and simulated using MultiBatchDS.

Example 1

A mixture containing 100 mole of a three-component mixture having relative volatilities of 1.7, 1.16, 1.0 is to be distilled in a batch distillation column. The column has 10 theoretical plates with a hold-up of 2 mol/plate and a condenser hold-up of 2 mol. The vapor boil-up rate V of the reboiler is 100 mol/h. Assume initial total reflux condition and use the steady-state model to obtain the initial values for integration. The column is operating under a constant reflux mode with a reflux ratio equal to 2.0.

1. Simulate a 1-h operation of the column using a second-order Runge-Kutta method and compare the distillate composition profile with those obtained using the stiff integrator LSODE (MultiBatchDS). Use a step size of 0.01 h for the Runge-Kutta method.
2. Reduce the plate hold-up and condenser hold-up from 2 mol to 0.5 mol/plate. Is the Runge-Kutta method good for simulating the column under this condition?
3. Now reduce the plate hold-up and condenser

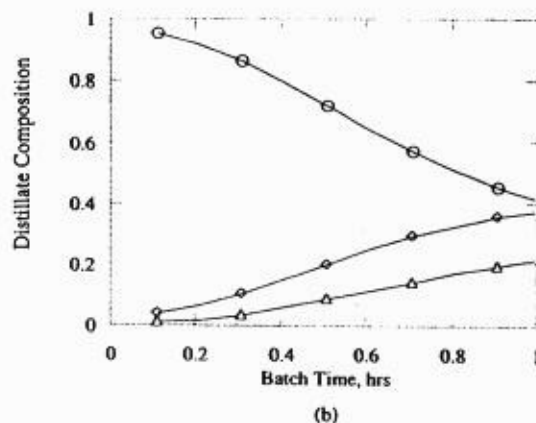
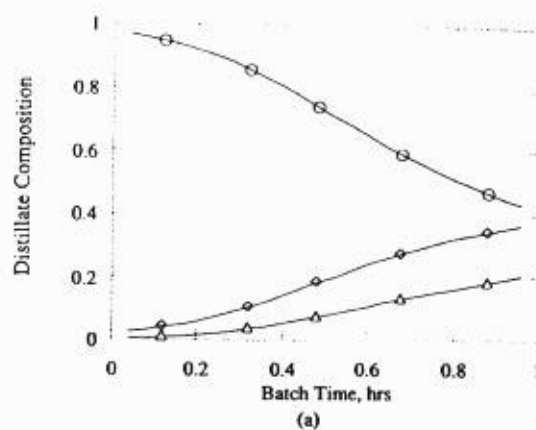


Figure 3 Transient composition profiles for Example 1.1 using (a) the second-order Runge-Kutta method and (b) the stiff integrator LSODE (MultiBatchDS).

holdup from 0.5 mol to 0.005 mol/plate and simulate the operation. Note the results.

Solution

1. Figure 3 shows the comparison of distillate composition profiles obtained using the two methods. The results indicate that the second-order Runge-Kutta method performs equally as well as the stiff integrator.
2. For the reduced-hold-up column, the results of the two methods are plotted in Figure 4. In this case, the two methods show significantly different results. Furthermore, the Runge-Kutta method results also show negative compositions as well as compositions exceeding unity, both of which are physically infeasible. One can conclude that owing to the stiff nature of the problem, the second-order Runge-

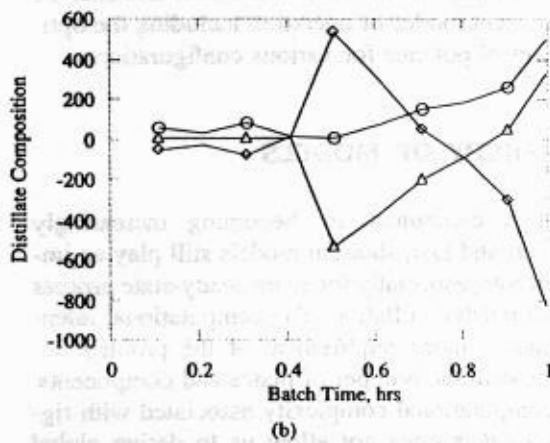
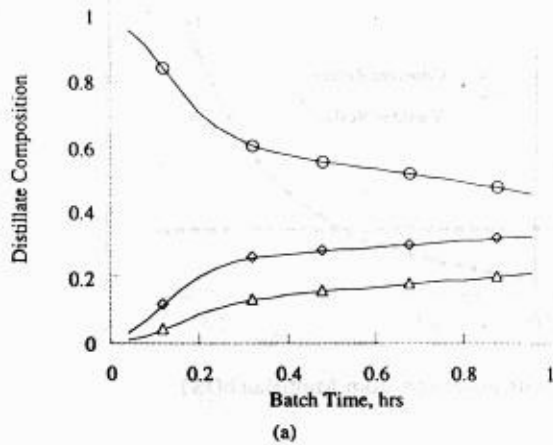


Figure 4 Transient composition profiles for Example 1.2 using (a) the second-order Runge-Kutta method and (b) the stiff integrator LSODE (MultiBatchDS).

Kutta method is not appropriate. This suggests the use of stiff integrators like LSODE for the solution of this problem. Figure 4 also shows the results of the batch distillation dynamics correctly represented using the LSODE integrator. Most of the batch distillation columns have hold-ups ranging from 5 to 10% and hence need stiff integrators to simulate the column dynamics. MultiBatchDS uses LSODE to simulate the rigorous column dynamics.

- Figure 5 shows the transient profiles obtained using LSODE. The LSODE method could not integrate the column, because the step size became so small that rounding errors dominated the performance.

The above example demonstrates that for columns where plate dynamics are significantly faster than reboiler dynamics (because of very small plate hold-ups), the stiff inte-

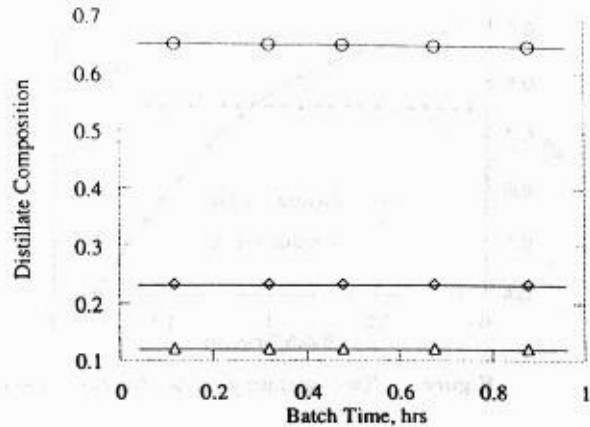


Figure 5 No solution from LSODE for Example 1.3.

grators often fail to find a solution. The solution to this problem is to split the system into two levels: (1) the reboiler, where the dynamics are slower, can be represented by differential equations; and (2) the rest of the column can be assumed to be in the quasi-steady state. This results in a zero hold-up model. This approach is used for a semirigorous model of MultiBatchDS. Figure 6 shows the results of this model. The model results are closer to those obtained previously in 1.1 and 1.2. The hold-up effects can be neglected in a number of cases where this model accurately approximates the column behavior. However, note that this model involves an iterative solution of nonlinear plate-to-plate algebraic equations, which can be computationally less efficient than the dynamic model. This low-hold-up, semirigorous model is used in many studies such as those of Domench and Enjal-

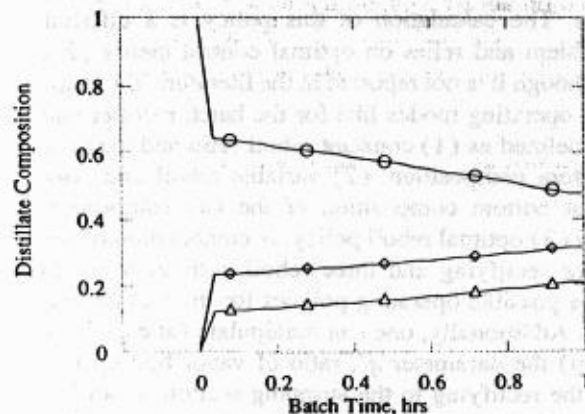


Figure 6 Transient composition profiles for Example 1.3 by switching the model.

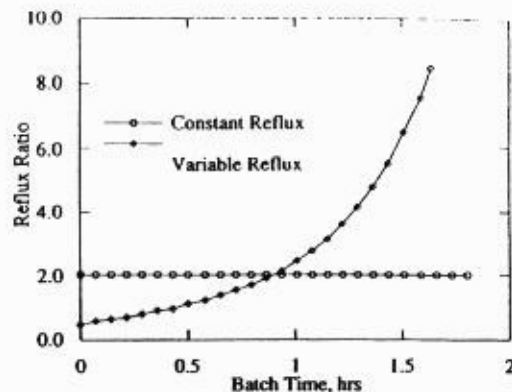
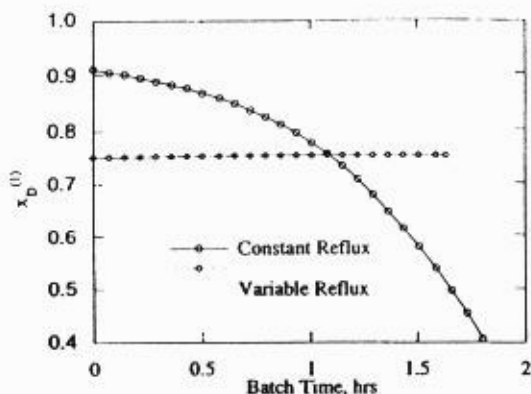


Figure 7 Two operating modes for batch rectifier (results generated from MultiBatchDS).

bert [9], Doherty and co-workers [10,11], and Farhat et al. [12].

MultiBatchDS provides the rigorous and semirigorous models for simulation of various column configurations described earlier and for simulation of different operating modes described in the next section.

DIFFERENT OPERATING POLICIES

The two basic modes of operating batch rectifying columns are (1) constant reflux and variable distillate composition, and (2) variable reflux and constant distillate composition of the key component (Fig. 7). There is a third operating mode of a batch rectifying column, which is neither variable reflux nor constant reflux, but is between the two. This type of operation is known as optimal reflux or optimal control policy. The optimal reflux policy is essentially a trade-off between the two operating modes of constant reflux and variable reflux, and is based on ability to yield the most profitable operation. The calculation of this policy is a difficult problem and relies on optimal control theory [2]. Although it is not reported in the literature, the stripper operating modes like for the batch rectifier can be defined as (1) constant reboil ratio and variable bottom composition, (2) variable reboil and constant bottom composition of the key component, and (3) optimal reboil policy. A combination of the three rectifying and three reboil policies result in nine possible operating policies for the middle vessel. Additionally, one can manipulate (at each time step) the parameter q' , ratio of vapor boil-up rate in the rectifying to the stripping section, as an important parameter for the middle vessel. With

MultiBatchDS it is possible to simulate and analyze the different modes of operation including the optimal control policies for various configurations.

HIERARCHY OF MODELS

Although computers are becoming increasingly powerful and fast, shortcut models still play an important role, especially for an unsteady-state process such as batch distillation. The computational intensity and memory requirement of the problem increases with the number of plates and components. The computational complexity associated with rigorous models does not allow us to derive global properties, such as feasible regions of operation, which are critical for simulation, design optimization, optimal control, and synthesis problems. Even if such information were available, the computational costs of optimization, optimal control, or synthesis using rigorous models is prohibitive. Therefore, MultiBatchDS offers a hierarchy of models ranging from the simplified model based on the shortcut method to the rigorous model based on Distefano analysis.

The shortcut method for batch distillation is based on the assumption that the batch distillation column can be considered a continuous distillation column with changing feed at any time instant. In other words, the bottom product of one time step forms the feed for the next time step. This is equivalent to having the bottom plate as the feed plate and the feed at its boiling point. Since continuous distillation theory is well developed and tested, the shortcut method [Fenske–Underwood–Gilliland (FUG) method] for continuous distillation can be modified for batch distillation. The batch time is implicit and can be calculated if the vaporization

rate V , of the reboiler is known. The shortcut method has been found to be extremely fast. Because of the noninteger (number of theoretic plates can be a noninteger variable) algebraic equation oriented from of the shortcut method, it is possible to adapt the model for optimal control and optimization calculations very easily. The details of developing the shortcut method for different operating modes of a rectifier are described elsewhere [2].

With the shortcut method it is possible to extract global properties of the batch column in terms of feasible region or inequality constraints. This is especially true for design variables like the number of plates, N , and the reflux ratio, R . This is a very useful property of the shortcut method. The identification of bounds on design parameters is also handy in design, optimization, or control problems. MultiBatchDS supports the design, optimization, and control options.

The shortcut method is applicable to nearly ideal (or ideal azeotropic) systems with a constant molal overflow assumption and columns with negligible hold-up effects. It has a minimum number of differential equations and a few algebraic equations to describe the complete dynamics. The next simplified model in the hierarchy is the reduced-order model based on the collocation approach.

The collocation approach was first proposed in the context of continuous-staged separation processes by Chao and Joseph [13,14] and followed

by a number of articles on the topic by Stewart et al. [15] and Srivastava and Joseph [16–18]. Diwekar [19] extended this approach to obtain the reduced-order model for batch distillation. The collocation approach to model reduction is based on approximating the column stage variables using polynomials rather than a discrete function of stages. Since the order of the orthogonal polynomials is less than the number of stages, the number of mass and energy-balance differential equations are less than those in the rigorous model. Apart from model reduction, this approach, like the shortcut method, can be used for a noninteger number of theoretic plates. Furthermore, the continuity of the state variables with respect to the height (instead of discrete equations at each equilibrium plate of the column) makes this model readily adaptable to packed columns.

PROGRAM STRUCTURE

MultiBatchDS is a very user-friendly package. It has a graphic interface for input and output that runs on Windows, Windows 95, and Windows NT. The input and output menus are easy to use and are very flexible. On-line help screens are available for each main menu, submenu, and input variable. It is also possible to monitor and control the program execution. For example, Figure 8 shows the main menu

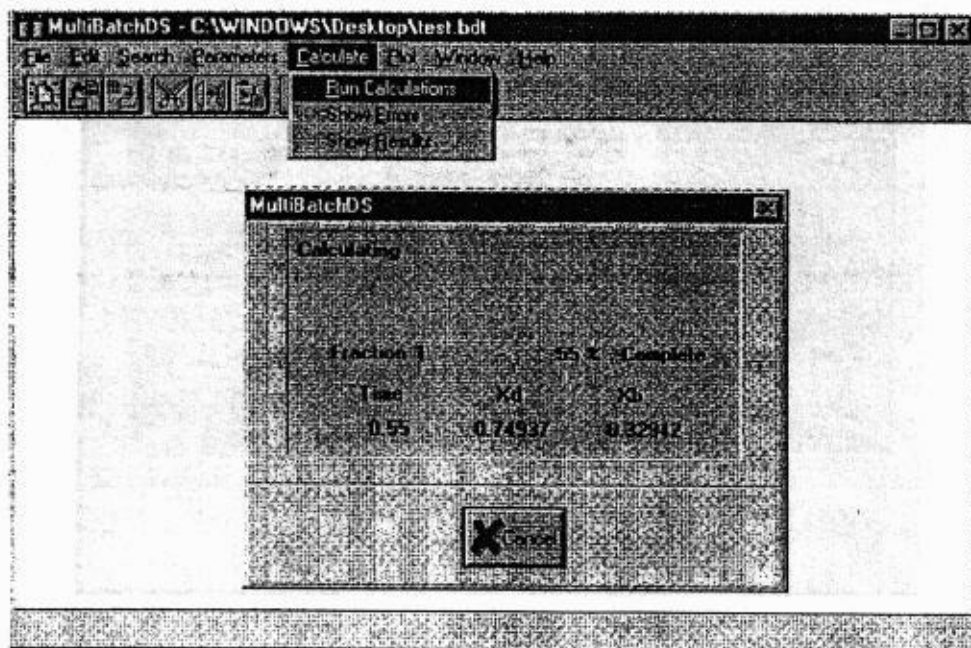


Figure 8 Windows-based input interface for MultiBatchDS.

Table 1 A Sample Keyword Input

```
C*SAMPLE KEYWORD INPUT FILE;
Policy or the operating mode = constant reflux;
Component-Code = 5,6,7;
Thermodynamic-Models = 0,0;
Task = Rigorous Simulation;
Column = Rectifier;
Feed-composition = 0.33,0.33,0.34;
Reflux-ratio = 1.0;
Number-of-Internal-Stages = 5;
Pressure-of-column in atms = 1.0;
Feed-Temperature in degrees c = 70.0;
Vapor-rate in moles/hr = 100.0;
Feed in moles = 100.0;
Batch-Time in hrs = 1.0;
Number-of-Fractions = 1;
Plate-Holdup in moles = 1.0;
Condenser-Holdup in moles = 1.0;
Initial-reflux = total;
End-Task = 1;
End-Policy = 1;
```

of the MultiBatchDS where the calculate menu is used to run the program. The smaller window shows the on-line simulation execution in terms of transient distillate and still composition for the key component. The cancel button in the smaller window can be used to stop the execution at any time.

MultiBatchDS also supports a keyword input structure in which commands can be written in simple English. The user can either use the graphic interactive input or the textual input through a batch file. Table 1 shows a sample keyword input file.

The output results are available in graph as well as text form. Figure 9 shows the plot menus for obtaining output results in various graph plots. It can be seen that the menu provides the flexibility of plotting different variables on abscissa and ordinates for multiple fractions.

MultiBatchDS could easily be linked to commercially available or in-house data banks. A wide variety of thermodynamic models are included in the package for calculating enthalpies, liquid densities, and vapor and liquid fugacities. However, the principles of the batch distillation process are more easily understood through simple thermodynamic models. Therefore, the educational version of the package comes with a constant relative volatility model, as well as a data bank of 103 commonly used chemical components. For components in the data bank one can choose the ideal fugacity models or the models based on the UNIFAC method (one parameter, two parameters, and three parameters) for non-ideal fugacity calculations. The data bank was provided courtesy of Dr. Gani in the Department of Chemical Engineering at the Technical University of Denmark.

A SIMPLE EXAMPLE

The following example illustrates how this package can be used to teach simple principles involved in batch distillation theory which are proposed from time to time by different researchers.

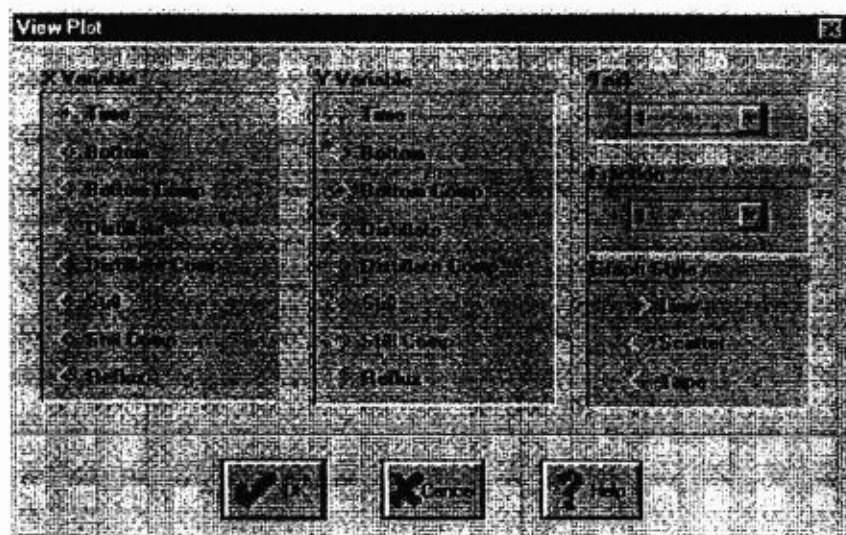


Figure 9 Plot menus for output results.

SEMI-RIGOROUS SIMULATION (CONSTANT REFLUX)

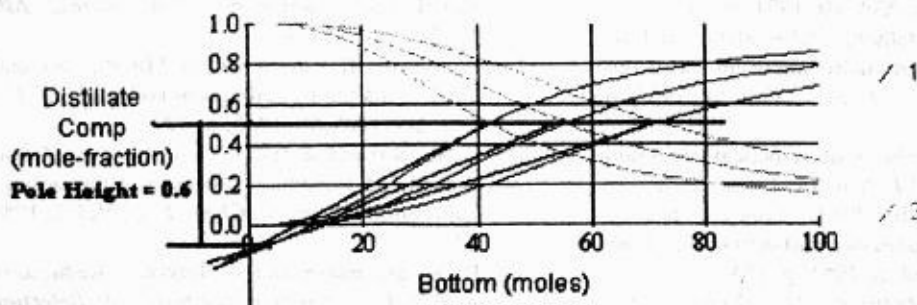


Figure 10 Solution plots from MultiBatchDS illustrating the pole height concept.

Example 2

In 1949, Bowman and Cichelli [20] presented a very interesting concept of pole height for a binary batch distillation column. Pole height is defined as the product of the midpoint of the slope of the distillate composition versus material remaining in the still curve, and the amount of the material remaining in the still at that time. They stated that the pole height is invariant to the initial concentration and provides a good measure for defining sharpness of separation.

Take 100 mol of a binary mixture containing component *A* and *B* with a relative volatility of 1.5. Use a five-theoretical stage batch distillation column and a constant reflux operation with a reflux = 5.0. Vary the initial composition from $x_A = 0.5$ –0.4 and plot the distillate composition versus the amount remaining in the still. Calculate the pole height for each case. Verify the concept.

Solution. The outputs (Fig. 10) from MultiBatchDS prove the validity of the pole height concept.

CONCLUSIONS

This article has described the features of the MultiBatchDS software package. The package is a handy tool to enhance the teaching of the physics and numeric principles involved in batch distillation in particular and batch processes in general. The constant relative volatility thermodynamics with binary systems and the semirigorous model (plate-to-plate calculations) can be used effectively in an undergraduate design course to explain the basics of batch distillation operations. This package can also serve as a learning aid for graduate courses. Furthermore, the students will find this package ex-

tremely useful in exploring new areas in batch distillation and batch process design research. A few examples of research projects which would be possible using this package include batch distillation synthesis, multiple column configurations, nonideal heterogeneous mixtures and their effect on dynamics, numeric stability, multivariable control, dynamic optimization, and scheduling. Currently we are also using this package to study the fugitive emissions problem with uncertainties.

ACKNOWLEDGMENTS

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REFERENCES

- [1] U. M. Diwekar and K. P. Madhavan, "BATCH-DIST: A comprehensive package for simulation, design, optimization, and optimal control of multicomponent, multifraction batch distillation columns," *Comput. Chem. Eng.*, Vol. 15, 1991, p. 833.
- [2] U. M. Diwekar, *Batch Distillation: Simulation, Optimal Design and Control*, Taylor and Francis International Publishers, Washington, DC, 1995.
- [3] A. G. Devidyan, V. N. Kiva, G. A. Meski, and M. Morari, "Batch distillation in a column with a middle vessel," *Chem. Eng. Sci.*, Vol. 49, 1994, p. 3033.
- [4] S. Skogstestad, B. Wittgens, E. Sorensen, and R. Litto, "Multivessel batch distillation," paper presented at the AIChE Annual Meeting, Miami, FL, 1995.
- [5] J. R. Gonzalez-Velasco, M. A. Gutierrez-Ortiz, J. S. Catresana-Pelayo, and J. A. Gonzalez-Marcos, "Im-

- provements in batch distillation startup," *Ind. Eng. Chem. Res.*, Vol. 26, 1987, p. 745.
- [6] W. L. Luyben, "Some practical aspects of optimal batch distillation design," *Ind. Eng. Chem. Process Design Development*, Vol. 10, 1971, p. 54.
- [7] G. P. Distefano, "Mathematical modeling and numerical integration of multicomponent batch distillation equations," *AIChE J.*, Vol. 14, 1968, p. 190.
- [8] G. P. Distefano, "Stability of numerical integration techniques," *AIChE J.*, Vol. 14, 1968, p. 946.
- [9] S. Domench and N. Enjalbert, "Program for simulating batch rectification as a unit operation," *Comput. Chem. Eng.*, Vol. 5, 1981, p. 181.
- [10] C. Bernot, M. F. Doherty, and M. F. Malone, "Patterns of composition changes in multicomponent batch distillation," *Chem. Eng. Sci.*, Vol. 45, 1990, p. 1207.
- [11] C. Bernot, M. F. Doherty, and M. F. Malone, "Feasibility and separation sequencing in multicomponent batch distillation," *Chem. Eng. Sci.*, Vol. 46, 1991, p. 1311.
- [12] S. Farhat, M. Czernicki, L. Pibouleau, and S. Domench, "Optimization of multiple-fraction batch distillation by nonlinear programming," *AIChE J.*, Vol. 36, 1990, p. 1349.
- [13] Y. S. Chao and B. Joseph, "Reduced-order steady-state and dynamic models of separation processes: Part I. Development of the model reduction procedure," *AIChE J.*, Vol. 29, 1983, p. 261.
- [14] Y. S. Chao and B. Joseph, "Reduced-order steady-state and dynamic models of separation processes: Part II. Application to nonlinear systems," *AIChE J.*, Vol. 29, 1983, p. 270.
- [15] W. Stewart, K. Levien, and M. Morari, "Simulation of fractionation by orthogonal collocation," *Chem. Eng. Sci.*, Vol. 40, 1985, p. 409.
- [16] R. K. Srivastava and B. Joseph, "Simulation of packed-bed separation processes using orthogonal collocation," *Comput. Chem. Eng.*, Vol. 8, 1984, p. 43.
- [17] R. K. Srivastava and B. Joseph, "Reduced-order models for separation columns—V: Selection of collocation points," *Comput. Chem. Eng.*, Vol. 9, 1985, p. 601.
- [18] R. K. Srivastava and B. Joseph, "Reduced-order models for staged separation columns—VI: Columns with steep and flat composition profiles," *Comput. Chem. Eng.*, Vol. 11, 1987, p. 165.
- [19] U. M. Diwekar, "Simulation, design, and optimization of multicomponent batch distillation columns," Ph.D. thesis, Indian Institute of Technology, Bombay, India, 1988.
- [20] J. R. Bowman and M. T. Cichelli, "Batch distillation: Minimum number of plates and minimum reflux," *Ind. Eng. Chem.*, Vol. 41, 1949, p. 1985.

BIOGRAPHY



Urmila Diwekar, Ph.D., is currently a senior research scientist in the Center for Energy and Environmental Studies and a research associate professor in the Department of Engineering and Public Policy at Carnegie Mellon University. She has published extensively in the areas of batch process modeling, dynamic optimization, process synthesis, and stochastic modeling and optimization. She has made major contributions to research on batch distillation, including authoring the first book concerning batch distillation, and this work is well recognized. Her academic research career is complemented by industrial experience, including consulting for several chemical engineering companies and national laboratories in the United States and overseas.