Study of the Separation of Simple Binary and Ternary Mixtures of Aromatic Compounds

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ABSTRACT: Consider binary and ternary mixtures of aromatic compounds composed of benzene/toluene and benzene/toluene/*p*-xylene, respectively. The present study shows how one can apply both mass and energy balance equations in order to understand the separation of such ideal mixtures by distillation. An adiabatic flash distillation problem is solved graphically for a mixture composed of benzene/toluene. Rigorous resolution of a steady-state binary distillation problem, using Mathematica® and the same benzene/toluene mixture, shows perfect agreement with results obtained using HYSYS. Results of a dynamic simulation involving the solution of a relatively large system of differential algebraic equations are presented and discussed for the benzene/toluene mixture. SIMULINK© and Mathematica® are used to perform control of this binary distillation column. Finally, a steady-state simulation of a simple multicomponent mixture, composed of benzene/toluene/*p*-xylene, is studied and some qualitative results are drawn from both the temperature and composition profiles. © 2011 Wiley Periodicals, Inc. Comput Appl Eng Educ, View this article online at wileyonlinelibrary.com/journal/cae DOI 10.1002/cae.20533

Keywords: distillation; Mathematica; dynamic and control, Ponchon–Savarit; aromatic mixtures

INTRODUCTION

The chemical engineering undergraduate program at King Fahd University of Petroleum & Minerals (KFUPM) is ABET accredited and offers two process separation courses. The junior course uses the textbook by Wankat [1] and introduces students to equilibrium-staged separations. A senior elective course is also given every other year and discusses more advanced distillation problems such as extractive and azeotropic distillations and uses the textbook by Geankoplis [2]. The department opted few years ago to phase out the computing laboratory, which trained students to use Matlab, HYSYS, and Excel in order to solve simple chemical engineering problems. Thus, every course in the new curriculum must train student to perform numerical computation using available software packages (i.e., solve problems related to their course with the help of computers). In this context a new experience was tested during fall 2010 in the junior separation course. Students were presented with the computer algebra Mathematica® and all homework assignments solutions were posted on the blackboard of KFUPM (called WebCT) in the form of Mathematica® notebooks. Many aspects of distillation such as flash operations, continuous column operation and control were introduced to students using homemade simulations with Mathematica®. Since distillation is a very important unit operation in the chemical industry and especially for the petroleum and petrochemical industry in the Kingdom of Saudi Arabia, students were keen to learn about this field and definitively adopted Mathematica® despite the fact that they have never been exposed to this computer package before. The present article describes a selection of study problems ranging from adiabatic flash distillation, binary distillation and multicomponent continuous distillation to dynamic behavior and control of binary distillation columns. All problems are treated with Mathematica® except the Wood and Berry control case study where SIMULINK® has clearly superior performances. The mixtures, to be separated by distillation, were intentionally exclusively taken from the aromatic compounds family (only ideal mixtures) in order to simplify computations such as the vapor–liquid and enthalpy calculations (i.e., students must know only about Raoult's law, simple mass and energy balance equations, basic liquid and vapor enthalpy calculations usually taught in the sophomore year at KFUPM). Important governing equations have been reported in the Appendix of the present article. All

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other equations are readily available from standard textbooks such as those written by Wankat [1] and Seader and Henley [3].

ADIABATIC FLASH DISTILLATION OF A BINARY MIXTURE

Consider a binary mixture composed of benzene and toluene. This mixture is fed to an adiabatic flash drum operating at 760 mmHg. The feed temperature and composition are taken equal to 240°C and 50 mol% of benzene (represented by a blue dot in Fig. 1). The feed is assumed to be liquid because it is initially at a very high pressure. Figure 1 plots the feed location in the enthalpy-composition or Ponchon–Savarit diagram as well as the vapor and liquid streams in equilibrium that exits the flash drum. The tie line (or isotherm) is also represented in this figure. The auxiliary line is represented and how this line can be utilized in order to get the tie lines. The compositions of the liquid and vapor phases leaving the flash drum are equal to 34.9 and 56.8 mol% benzene, respectively. The simulation use both energy and mass balance equations and allows the prediction of the temperature of the drum, which is equal for this particular case to 96.8°C. For this problem the vapor fraction is equal to 69.1%, which means that the liquid and vapor streams have flow rates equal to 69.1 and 30.9 kmol/h, respectively, assuming a feed flow rate of 100 kmol/h. Figure 2 shows the flash distillation setup. A neat feature of Mathematica® is the possibility to vary the simulation parameters using sliders and to get instantaneous results reported in the figures. This capability can be put into advantage in order to promptly show students various scenarios such as varying the feed thermal quality, by changing the feed's temperature, as well as the feed composition. The authors find the performance of Mathematica® for similar problems involving graphical resolutions to be

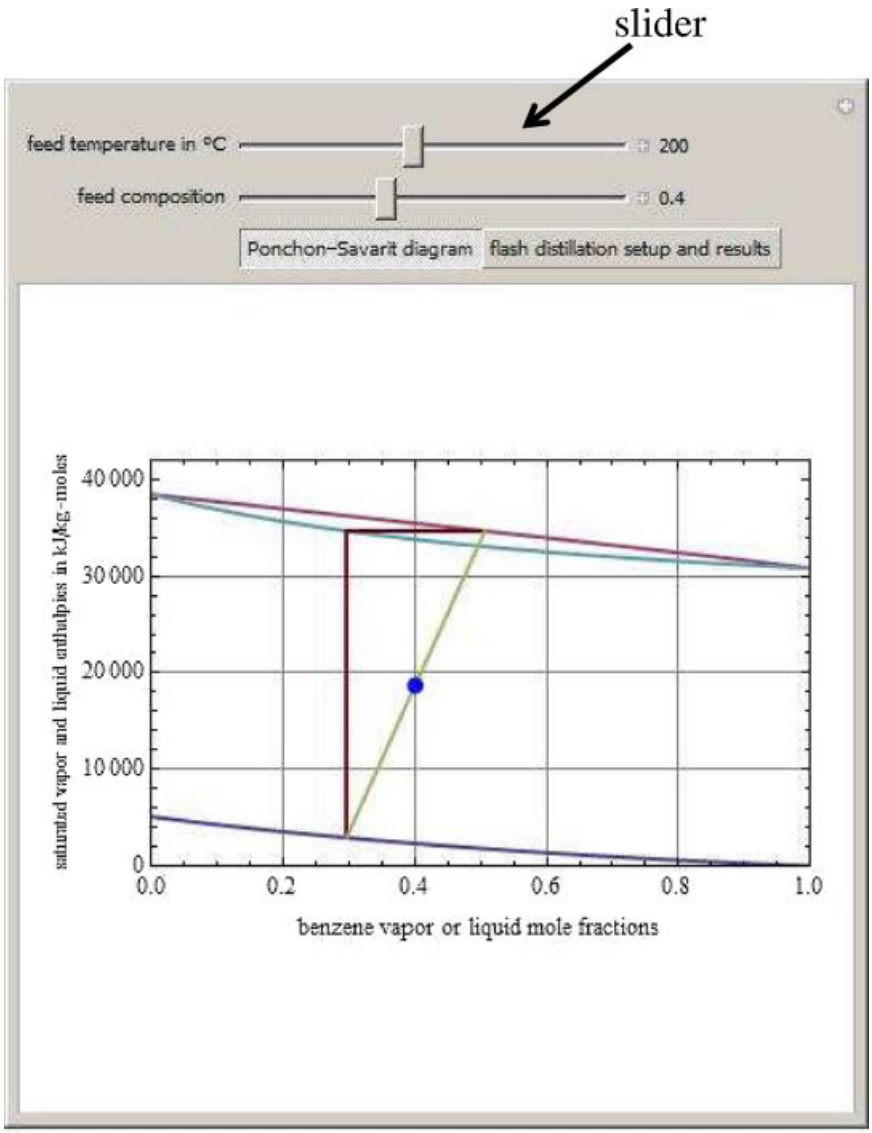

Figure 1 Ponchon–Savarit diagram shown isotherm and feed location. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

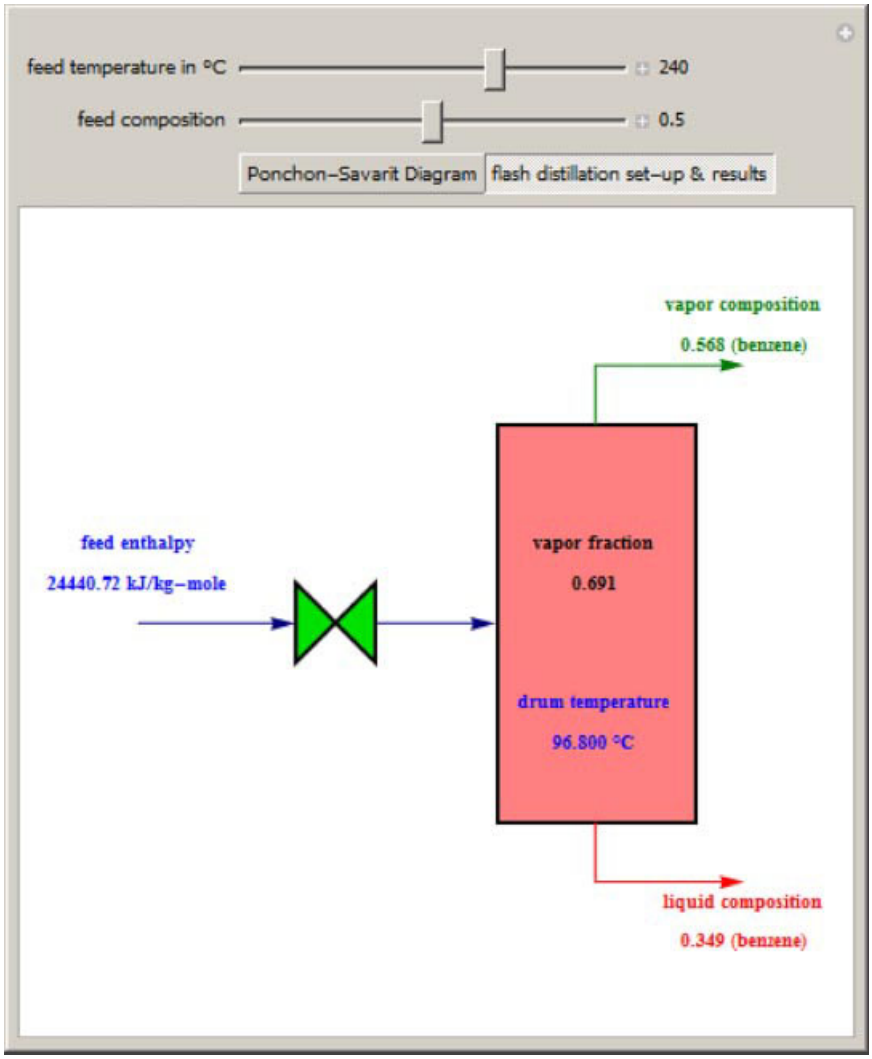

Figure 2 Flash distillation setup. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

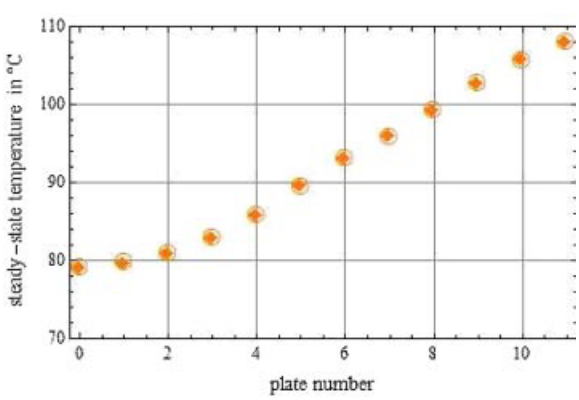

Figure 3 Temperature profile. HYSYS and Mathematica® results are shown by ○ and ◆, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

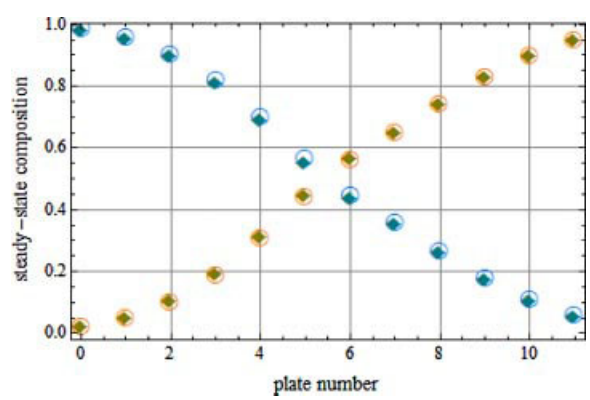

Figure 4 Composition profile for benzene (blue symbols) and toluene (brown symbols). HYSYS and Mathematica® results are shown by ○ and ◆, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

superior to other available pedagogical tools and methods such as looking up values of enthalpies from diagrams and doing trial-and-error calculations, which are often presented in most separation science textbooks.

RIGOROUS STEADY-STATE SIMULATION OF A BINARY DISTILLATION COLUMN

Consider an ideal equimolar binary mixture composed of benzene and toluene at 760 mmHg. This mixture, with a thermal quality equal to 0.5, is fed to a 10 stages column with total condenser and partial reboiler. Feed stage is plate number 7. Feed flow rate is taken equal to 10 kmol/h. Stage indexing is such that the top stage is stage number 1. Figures 3 and 4 show the temperature and composition profiles using a rigorous approach, which includes both the energy and mass balance equations. All results found in the present study show perfect agreement with those given by HYSYS (http://www.aspentech.com/hysys/), which are also indicated in the composition and temperature profiles. Distillate and bottom purities are found equal to 98.2 and 5.41 mol% benzene, respectively. Figure 5 shows that the numerical simulation gives data similar to the graphical method developed by Ponchon–Savarit. Difference point coordinates are (0.982, 123527) and (0.0541001, −79362) for the rectifying and stripping sections, respectively. The molar flow rate profiles for both the liquid and vapor phases are also displayed in Figure 6. Non Constant Molal Overflow calculation is justified by the fact that these flow rates are not constant in both the rectifying and the stripping sections of the column. Calculations show that for $R = 3$ and $S = 2.5$, stage 7 is the optimal feed location. Here, $R = L/D$ and $S = V/B$ are the reflux and reboil ratios, respectively. Indeed, if one changes the feed location, while keeping the same values for R and S, the cooling and heating duties will be higher (see Table 1). It is the belief of the authors that optimal feed location is a

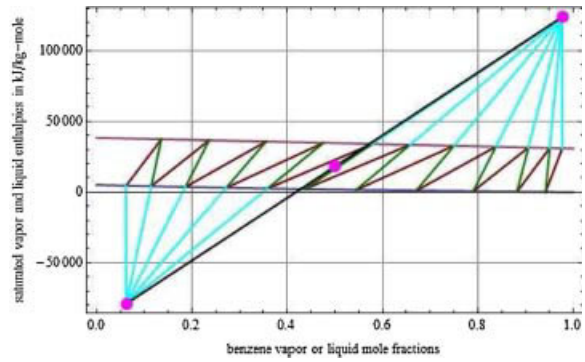

Figure 5 Ponchon–Savarit diagram. Magenta dots are difference points. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

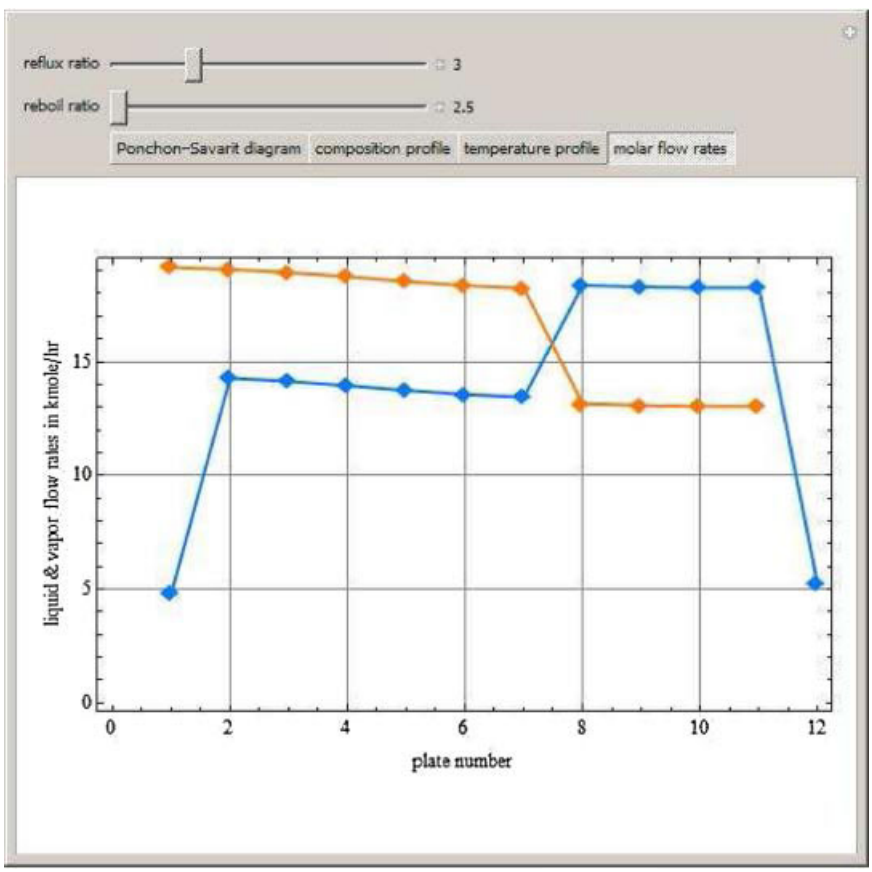

Figure 6 Profile for liquid and vapor flow rates in blue and orange, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table 1 Heat Duties Versus Feed Plate Location

Feed location	Q_c (kJ/h)	Q_r (kJ/h)
Stage 3	1089308	977764
Stage 4	594638	436504
Stage 5	595341	436892
Stage 6	594308	436317
Stage 7	**594284**	**436303**
Stage 8	594688	436532
Stage 9	1094228	980764

Values in bold correspond to the optimal feed stage case.

concept difficult to grasp by students unless some kind of numerical proof is given, which can be done in class in a matter of minutes using our Mathematica® simulation. For this example, we have found cooling and heating duties equal to $Q_c = -594284$ kJ/h and $Q_r = 436303$ kJ/h, respectively, for $R = 3$ and $S = 2.5$. A simple observation of the Ponchon-Savarit diagram confirms that optimal feed plate location is stage 7 since the line joining the operating points (shown by magenta dots) and the feed point (indicated with a red dot) is just after stage 7. If one takes a close look at the Ponchon–Savarit diagram and applies the lever-arm rule, he will clearly see that the feed's vapor fraction is 50%. Study of this binary distillation problem involves solving a system of 60 nonlinear algebraic equations, which is done in a matter of a fraction of a second using Mathematica®. Thus, such calculations using any computer algebra along with the simulation using HYSYS can be readily introduced in two 1-h lectures to junior or senior students in a separation science course. This teaching strategy was tested successfully at KFUPM during the fall semester of 2010. The possibility to move around sliders (see Fig. 6) and select various values of the reboil and reflux ratios are also put into advantage in this problem to discuss issues such as optimal

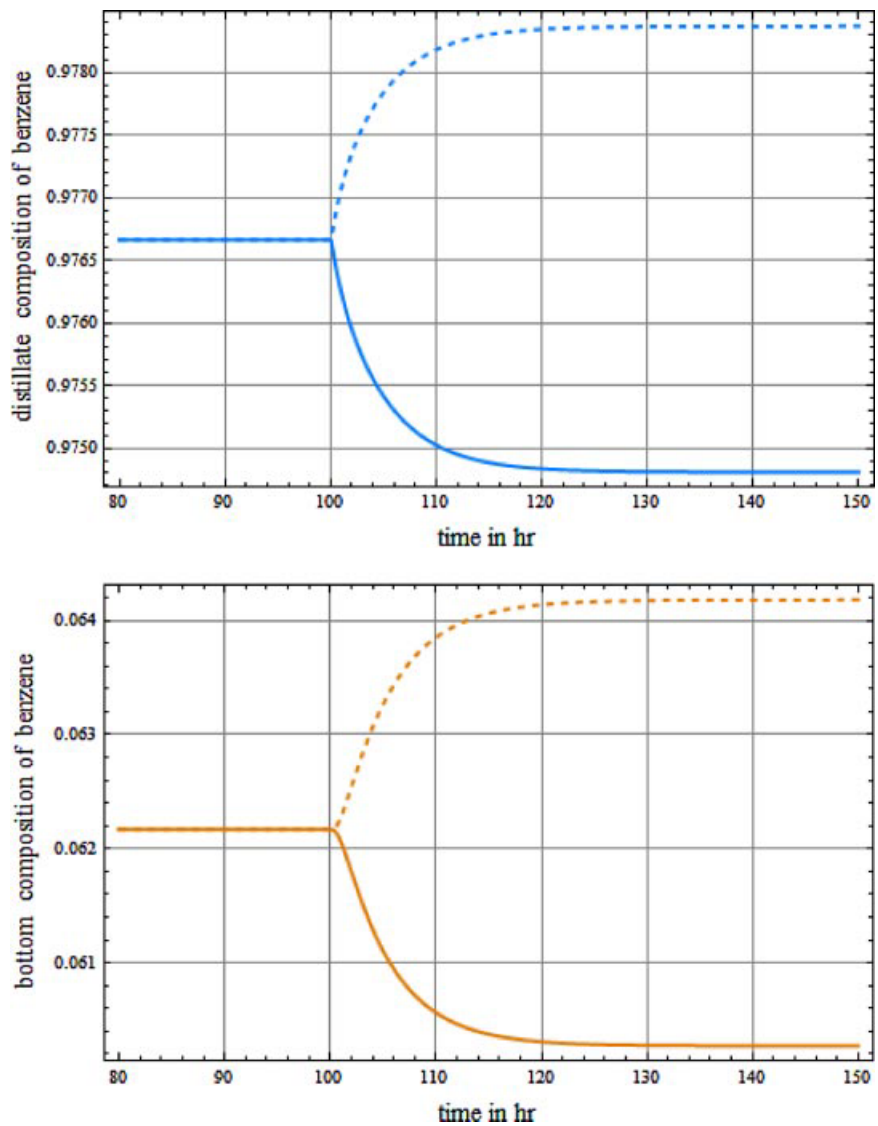

Figure 7 Behavior of distillate and bottom composition for a +1% step (dashed curve) and −1% step (continuous curve) change in the reflux ratio. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

feed location, bottom and distillate compositions and difference point positions (shown with magenta dots in Fig. 5). Finally, how Ponchon–Savarit construction is built by alternating between operating (cyan and green segments) and tie lines (red segments) can be readily understood by students as depicted in Figure 5.

DYNAMIC SIMULATION OF A BINARY DISTILLATION COLUMN

Consider an ideal equimolar binary mixture of benzene and toluene at 760 mmHg. This mixture, with a thermal quality equal to 0.5, is fed to a 10-stage column with a total condenser and partial reboiler. The feed stage is again Plate 7. The feed flow rate is taken equal to 10 kmol/h. Up to time $t = 100$ h, the reflux and reboil ratios are $R = 3$ and $S = 2.5$, respectively. At times above 100 h, the column is subject to different scenarios such as a step, $\pm 1\%$, in the reflux ratio, R, in the reboil ratio, S, or in the feed composition. A rigorous simulation using Mathematica® (i.e., using both mass and energy balance equations) computes the dynamic behavior of the distillate and bottom compositions (e.g., benzene mole fractions) for each scenario. It can be seen that an increase in the reflux ratio ($+1\%$ step) causes a simultaneous increase in distillate purity at the expense of a decrease in bottom composition purity. On the other hand a $+1\%$ step in the reboil ratio has the inverse effect (i.e., an increase in bottom purity at the expenses of distillate quality). For the purpose of simplicity, the molar holdups of the condenser, reboiler, and plates are assumed constant, and equal to 5, 10, and 1 kmol, respectively. As an alternative and more rigorous route, one could easily include in the code the Francis weir formula in order to compute time-dependent molar holdups. The results of the simulation are shown in Figures 7–9.

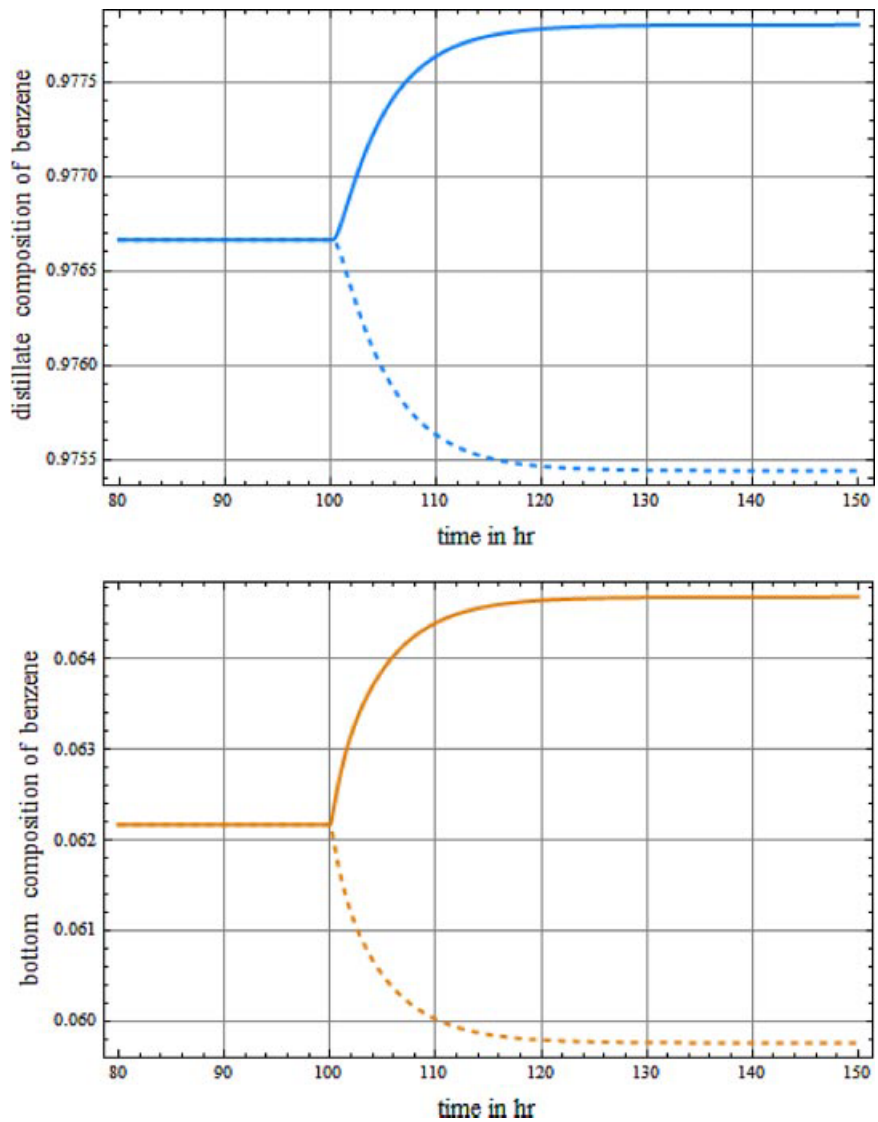

Figure 8 Behavior of distillate and bottom composition for a $+1\%$ step (dashed curve) and -1% step (continuous curve) change in the reboil ratio. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

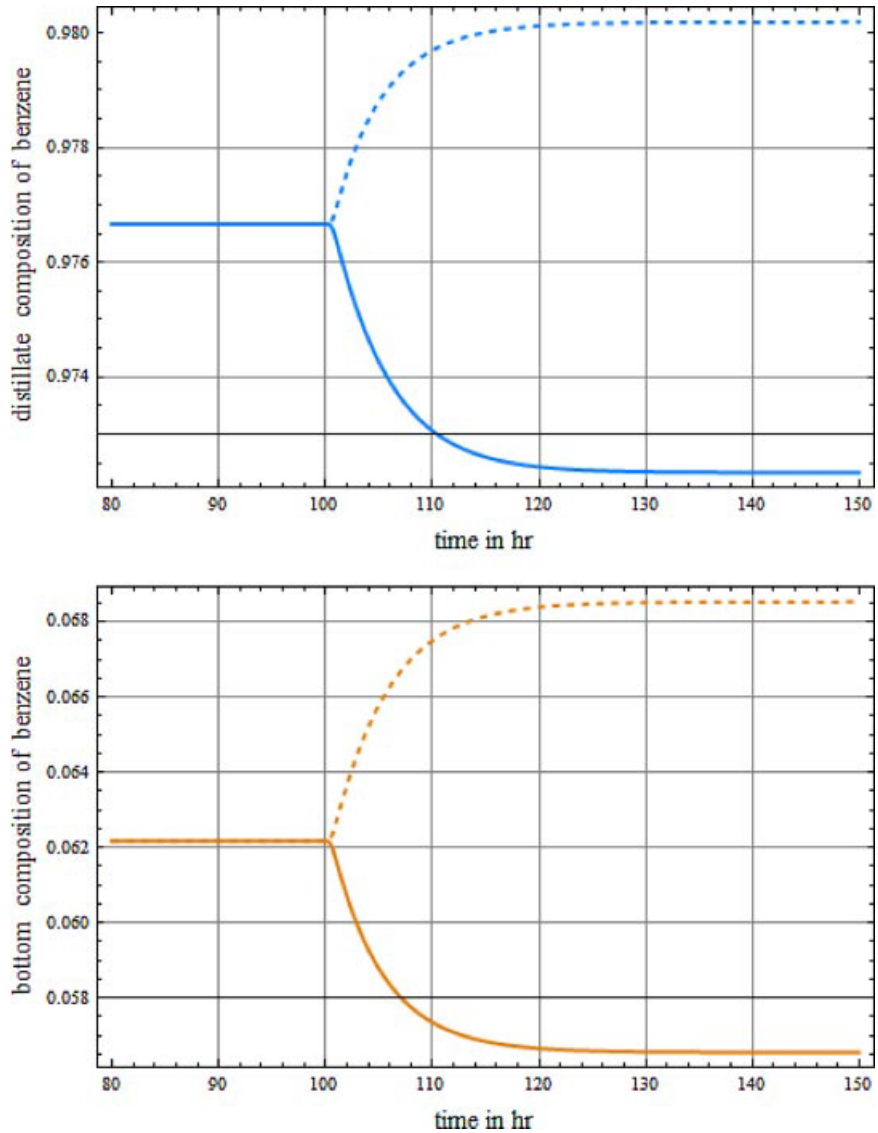

Figure 9 Behavior of distillate and bottom composition for a +1% step (dashed curve) and −1% step (continuous curve) change in the feed composition. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Here, the authors solve a system of 60 DAEs in a fraction of a second. First-order transfer functions with dead time, obtained also using Mathematica®, are given below:

$$G_{11}(s) = \frac{0.040e^{-0.02s}}{0.237 + s} \qquad G_{21}(s) = \frac{0.034e^{-0.3s}}{0.170 + s}$$

$$G_{12}(s) = \frac{-0.022e^{-0.3s}}{0.178 + s} \qquad G_{22}(s) = \frac{-0.058e^{-0.1s}}{0.243 + s}$$

$$G_{d1}(s) = \frac{0.069e^{-0.3s}}{0.196 + s} \qquad G_{d2}(s) = \frac{0.114e^{-0.3s}}{0.179 + s}$$

where the input–output model is:

$$\begin{bmatrix} x_d(s) \\ x_b(s) \end{bmatrix} = \begin{bmatrix} G_{11}(s) & G_{12}(s) \\ G_{21}(s) & G_{22}(s) \end{bmatrix} \begin{bmatrix} R(s) \\ S(s) \end{bmatrix} + \begin{bmatrix} G_{d1}(s) \\ G_{d2}(s) \end{bmatrix} [x_f(s)]$$

The outputs are the distillate and bottom compositions, x_d and x_b, respectively. The manipulated inputs are the reflux and reboil ratios, R and S, respectively. The disturbance input is the light component mole fraction, x_f.

CONTROL OF A BINARY DISTILLATION COLUMN

Dual product control of a binary distillation column, using reflux and reboil ratios as manipulated variables, is difficult because the two control loops interact. For example, if distillate purity needs to be adjusted, the reflux ratio is increased, which affects in a negative way the bottom purity. Thus, the reboil ratio is manipulated by the bottom control loop, which increases the overhead vapor flow rate and affects the top control loop. Wood and Berry [4] devised a non-interacting control

system based on the knowledge of the transfer functions. The results show a very significant improvement in the control of both distillate and bottom compositions as well as rejection of feed composition disturbances. Based on the above-mentioned non-interacting control method and on our binary distillation dynamic simulation (see previous section where we obtained all relevant transfer functions), the authors have developed a model using SIMULINK® (see Fig. 10). In Figures 11 and 12, the distillate and bottom compositions are depicted versus time when the column is subject to a +1% change in feed composition at $t = 0$ h and when the setpoints of the top and bottom product are 96 and 3 mol% benzene, respectively. The simulation uses simple PID controllers and the controller settings are given in Table 2. Another approach to the dual quality control of a distillation column is to use the control scheme

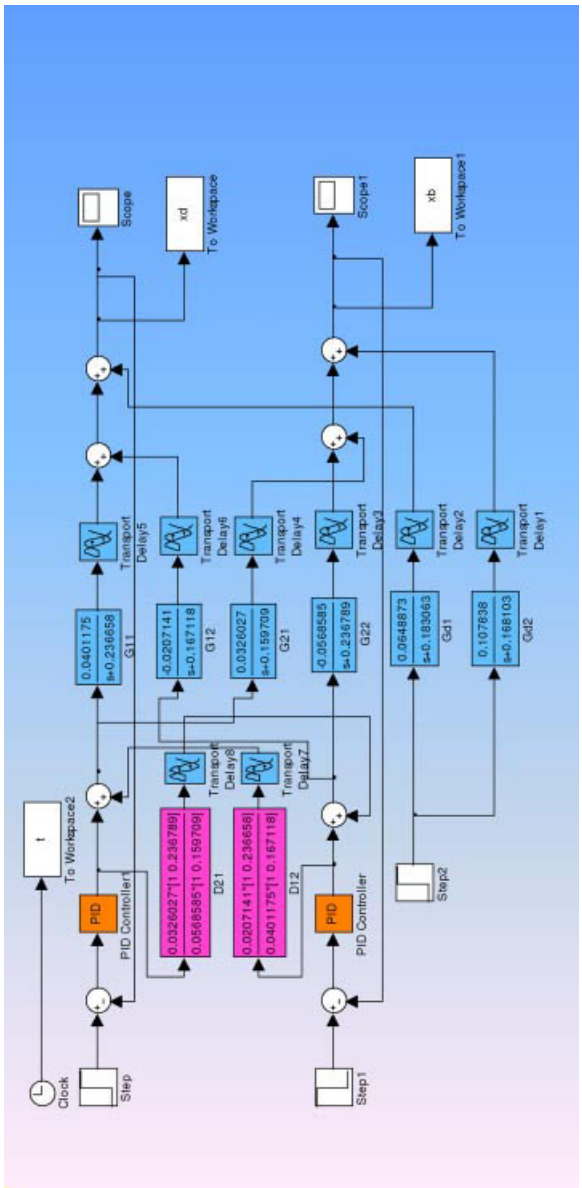

Figure 10 Simulink model for the Wood and Berry method.[Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

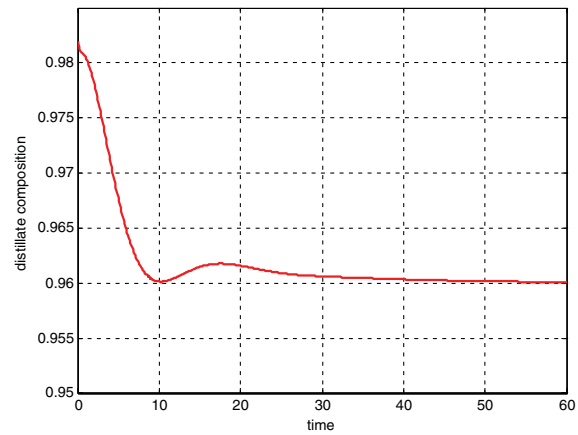

Figure 11 Distillate composition versus time (setpoint 96 mol% fraction). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

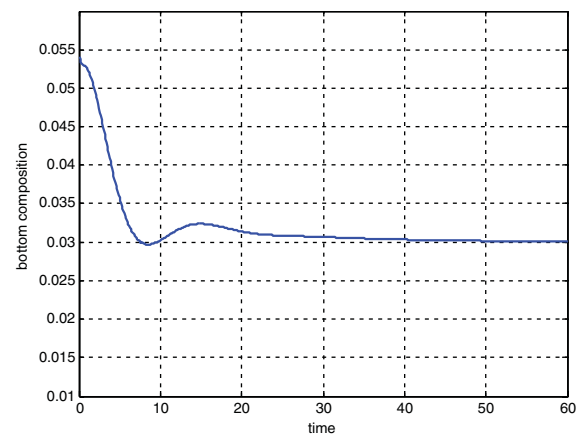

Figure 12 Bottom composition versus time (setpoint 3 mol% fraction). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

suggested by Rijnsdorp [5,6] and later by Rijnsdorp and Van Kampen [7]. This scheme, which can be easily implemented with Mathematica®, utilizes a ratio controller to control the ratio of the overhead vapor rate to reflux flow rate. The setpoint of the ratio controller (slave loop) is fixed by the distillate composition controller (master loop). Thus, distillate composition is no longer affected by variations in the reboil ratio, which cause the overhead vapor rate to change. Figures 13 and 14 show the distillate and bottom compositions when the setpoints are chosen equal to 96.0 and 3.0 mol% benzene, respectively. The reflux and reboil ratios versus time are depicted in Figure 15. For this particular case, final values of both ratios are higher as can be seen in Figure 15.

Table 2 Constants for PID Controllers Where $G_c(s) = K_p + (K_i/s)$

Top PID controller	Bottom PID controller
$K_p = 3$	$K_p = -3$
$K_i = 5$	$K_i = -5$

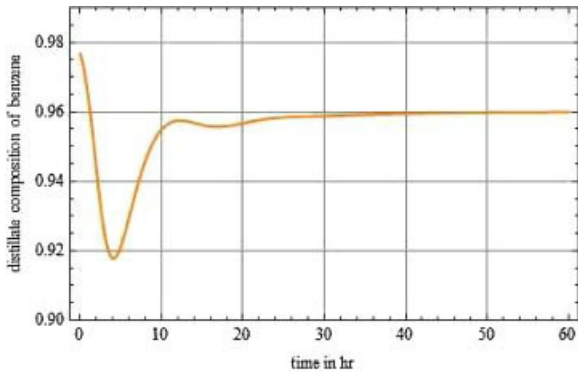

Figure 13 Distillate composition versus time (setpoint equals 96 mol% benzene). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

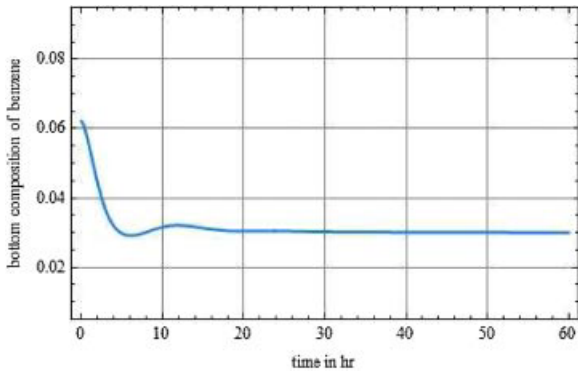

Figure 14 Bottom composition versus time (setpoint equals 3 mol% benzene). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

RIGOROUS SIMULATION OF A MULTICOMPONENT DISTILLATION COLUMN

Consider a ternary mixture composed of benzene (23.33 mol%), toluene (33.33 mol%), and *p*-xylene (43.34 mol%) at 101.325 kPa. This mixture, with a thermal quality equal to 1.0 (i.e., a saturated liquid), is fed to a 19-stage column with a total condenser

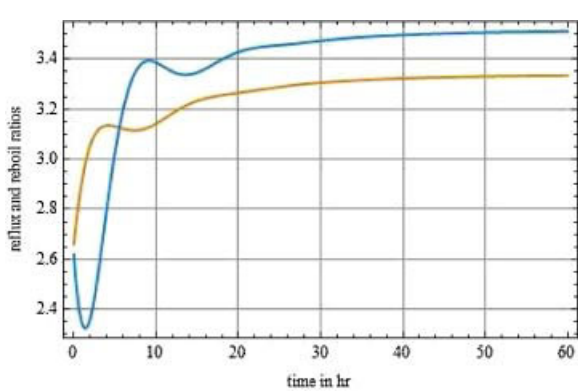

Figure 15 Behavior of the reboil (orange curve) and reflux (blue curve) ratios versus time. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

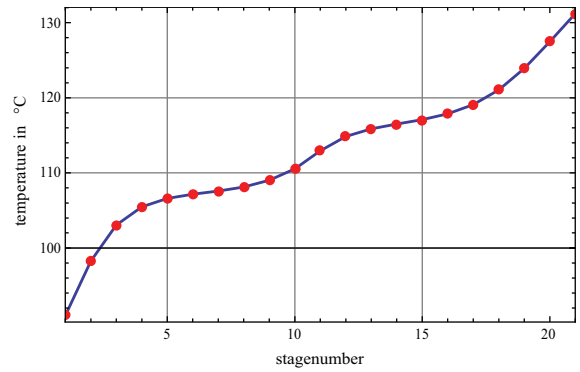

Figure 16 Temperature profile. HYSYS and Mathematica® results are shown by ● and −, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

(taken as stage number 1) and a partial reboiler (stage 21). The feed enters at stage 10. The feed flow rate is set equal to 1 kmol/h. One can compute the temperature (see Fig. 16) and composition (see Fig. 17) profiles using a rigorous approach, which includes both the energy and mass balances thanks to Mathematica®. The blue, magenta, and brown curves, shown in the composition profile, correspond to benzene, toluene, and *p*-xylene compositions, respectively. For $R = 4$ and $S = 4$, the results found in the present calculation using Mathematica® show perfect agreement with those given by HYSYS (http://www.aspentech.com/hysys/). HYSYS data are shown with colored dots. The temperature profiles for both binary (see Fig. 3) and ternary (see Fig. 16) mixtures are similar and exhibit a monotonic increase going from condenser to reboiler. On the other hand, for the composition profiles of the binary (see Fig. 4) and ternary (see Fig. 17) mixtures, there are very clear differences. Indeed, for the ternary system, the light-key, LK, and heavy-key, HK, are benzene and toluene, respectively. There is a heavy-non-key, HNK, which is *p*-xylene. The HNK is non-distributing and appears only in the residue. The HK component presents maxima in the composition profile while no maximum is present for composition profile of the binary separation case. All components must be present at the feed stage and there is a discontinuity in the composition profile at that stage. Finally, HNK composition goes through a plateau

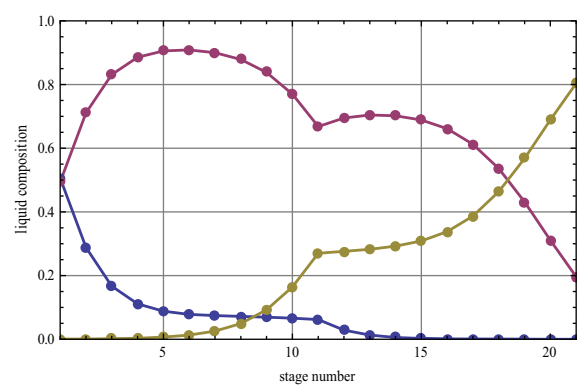

Figure 17 Composition profile for benzene (blue), toluene (magenta), and *p*-xylene (brown). HYSYS and Mathematica® results are shown by ● and −, respectively. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

region. Heat and cooling duties were found equal to $Q_c = -59817.1$ kJ/h and $Q_r = 60997.2$ kJ/h, respectively. The authors assumed constant values for the liquid and vapor heat capacities of benzene, toluene, and *p*-xylene, although the calculation of enthalpies can be improved by taking temperature-dependent heat capacities. Again, sliders allow instantaneous computation of composition and temperature profiles for any user-selected values of the reflux and reboil ratios as shown in Figure 18. The calculation for this particular ternary separation problem involves solving 147 nonlinear algebraic equations, which is done with Mathematica® in a fraction of a second.

RELEVANT Mathematica® COMMANDS

When compared to other numerical computation packages, Mathematica® has the least steep learning curve. Indeed, it is a matter of a couple of hours that one can learn to solve systems of algebraic equations using the built-in command *FindRoot*, to solve systems of differential and algebraic equations using the extremely powerful command *NDSolve* and to plot solutions with the commands *Plot* and *ListPlot*. Another useful function is *Fit*, which performs nonlinear curve fitting. Table 3 gives the basic functions employed to solve the various problems presented above. Students really enjoy discovering about this extraordinary computational tool and usually express their wish that they should be exposed to Mathematica® earlier in their academic curriculum.

CONCLUSION

The present study shows how one can employ state-of-the-art computer mathematical programs such as Mathematica® and SIMULINK® to learn about the separation of simple binary and ternary ideal mixtures composed of solely aromatic compounds (i.e., benzene, toluene, and *p*-xylene). Such calculations can be used in the classroom to illustrate several aspects related to flash distillation, continuous binary distillation (steady-state problems, dynamic behavior and good control schemes) and

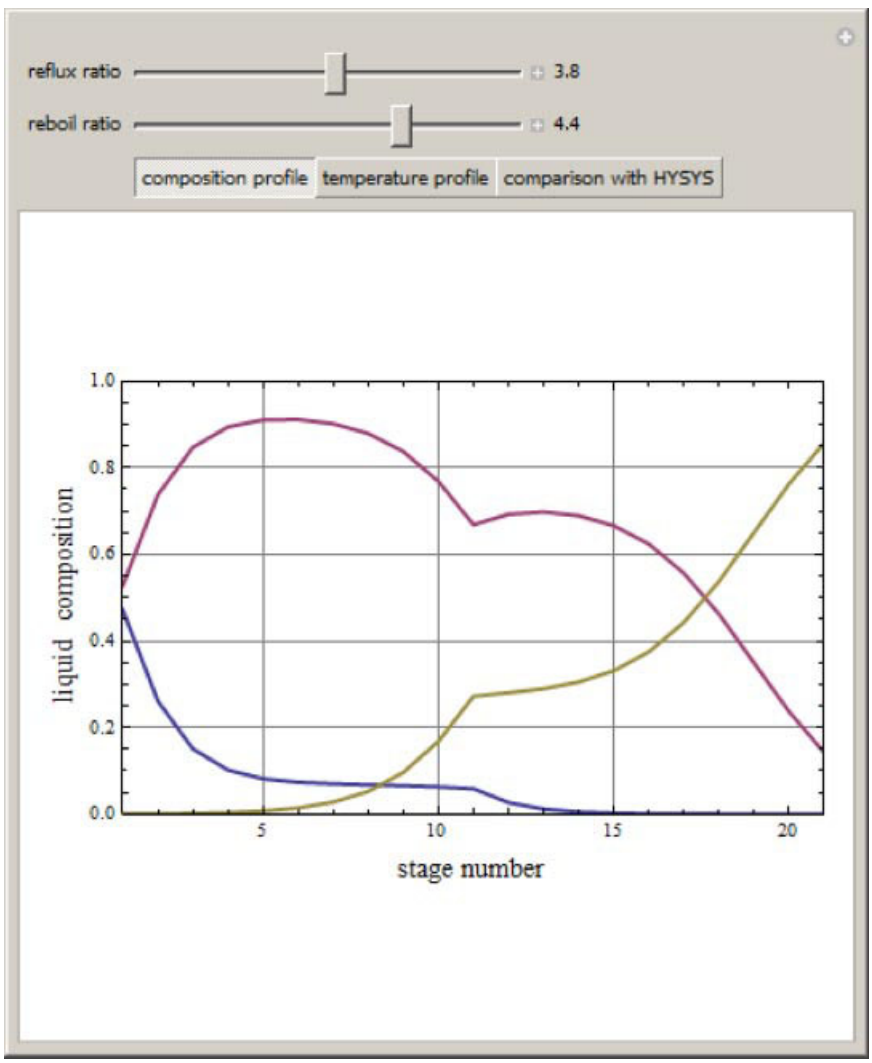

Figure 18 Sliders allowing instantaneous calculation if the reboil and reflux ratios are modified. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table 3 Mathematica® Commands

Mathematica® command	Chemical engineering problem
FindRoot (equivalent to fsolve with Matlab)	Steady-state distillation simulations involving algebraic mass and energy balance equations as well as algebraic VLE relationships. *FindRoot* uses Newton–Raphson as well as various other methods such as the secant method
NDSolve (equivalent to ode15s with Matlab)	Dynamic distillation simulations involving differential mass and energy balance equations as well as algebraic VLE relationships. *NDSolve* uses the IDA method, which is designed to generally solve index-1 DAEs, but may work for higher index problems as well
Plot and ListPlot (equivalent to plot with Matlab)	Graphical visualization of lists of data and functions
Fit and FindFit (equivalent to fit and polyfit with Matlab)	Curve fitting of experimental data using least squares as well as many other methods such as conjugate gradient, Levenberg–Marquardt ...
Manipulate (equivalent to guide with Matlab)	Generates expressions, figures or tables with controls added to allow interactive manipulation of the value of various variables

continuous multicomponent distillation. It is possible to extend the numerical simulations to include (1) non-ideal liquid phase behavior by using an appropriate activity coefficient model such as NRTL or Wilson ..., (2) high pressure effects using equations of state (EOS), such as the Peng–Robinson EOS and the Soave–Redlich–Kwong EOS, or the Hayden–O'Connell method, and (3) batch distillation problems. The authors hope that this contribution will provide to chemical engineering faculty in other institutions a new kind of study material, which is currently tested at KFUPM in the junior and senior separation science courses. The authors encourage readers to use the educational programs developed at KFUPM in their classes and all simulation notebooks can be obtained from the corresponding author upon request.

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APPENDIX

The governing equations that allow the steady-state and dynamic simulations to be performed are given below. For steady-state simulations the LHS term should be set equal to zero. They are composed of the total and partial mass balances and the energy balance around the feed stage, the partial reboiler, the total condenser and any tray other than the feed tray. These balance equations, written separately for all the column sections mentioned above, are the following:

(1) Feed tray ($k = f$):

$$\frac{dM_f}{dt} = F + L_{f-1} + V_{f+1} - L_f - V_f \tag{A1}$$

$$\frac{d(M_f x_{f,i})}{dt} = F z_{f,i} + L_{f-1} x_{f-1,i} + V_{f+1} y_{f+1,i} - L_f x_{f,i} - V_f y_{f,i} \text{ for } i = 1, 2, \ldots, N_c \tag{A2}$$

$$\frac{d(M_f h_f)}{dt} = F h_F + L_{f-1} h_{f-1} + V_{f+1} H_{f+1} - L_f h_f - V_f H_f \tag{A3}$$

(2) kth tray ($k \neq f$ and $1 < k < N$):

$$\frac{dM_k}{dt} = L_{k-1} + V_{k+1} - L_k - V_k \tag{A4}$$

$$\frac{d(M_k x_{k,i})}{dt} = L_{k-1} x_{k-1,i} + V_{k+1} y_{k+1,i} - L_k x_{k,i} - V_k y_{k,i} \text{ for } i = 1, 2, \ldots, N_c \tag{A5}$$

$$\frac{d(M_k h_k)}{dt} = L_{k-1} h_{k-1} + V_{k+1} H_{k+1} - L_k h_k - V_k H_k \tag{A-6}$$

(3) Reflux drum ($k = 1$):

$$\frac{dM_1}{dt} = V_2 - (L_1 + D) \tag{A-7}$$

$$\frac{d(M_1 x_{1,i})}{dt} = V_2 y_{2,i} - (L_1 + D) x_{D,i} \text{ for } i = 1, 2, \ldots, N_c \tag{A8}$$

$$\frac{d(M_1 h_1)}{dt} = V_2 H_2 - (L_1 + D) h_1 - \dot{Q}_C \tag{A9}$$

(4) Reboiler ($k = N$):

$$\frac{dM_N}{dt} = L_{N-1} - L_N - V_N \tag{A10}$$

$$\frac{d(M_N x_{N,i})}{dt} = L_{N-1} x_{N-1,i} - L_N\ x_{N,i} - V_N y_{N,i} \text{ for } i = 1, 2, \ldots, N_c \tag{A11}$$

$$\frac{d(M_N h_N)}{dt} = L_{N-1}\ h_{N-1} - L_N\ h_N - V_N\ H_N + \dot{Q}_B \tag{A12}$$

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