Simple Batch Distillation of a Binary Mixture

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ABSTRACT: A simple experiment such as the batch distillation of an ethanol–water binary mixture can be performed in a 3-h laboratory session using a very rudimentary apparatus consisting of a still pot and two thermocouples. Yet, the results can lead to very interesting insights to distillation and chemical engineering thermodynamics. The present article describes how one can exploit this simple laboratory experiment to teach these various aspects of chemical engineering. In addition, several simple calculations using computer software such as Mathematica® are performed to explain, interpret and reproduce theoretically all the gathered experimental data during the laboratory session.   © 2012 Wiley Periodicals, Inc. Comput Appl Eng Educ; View this article online at wileyonlinelibrary.com/journal/cae; DOI 10.1002/cae.21556

INTRODUCTION

In an attempt to introduce new or back-up experiments into the senior unit operations laboratory at King Fahd University of Petroleum and Minerals (KFUPM), CHE 409, the authors have performed a series of simple batch experiments using a binary mixture composed of ethanol and water. The aim of this work is to show to our students a time-dependent behavior, which is typical of batch experiments. Such an experiment can complement the already existing continuous distillation of an ethanol–water mixture laboratory, called UOP 2. Batch distillation is taught at KFUPM in a junior course, CHE 306. Three or four 1-h lectures are devoted to batch distillation using Chapter 9 of Wankat's textbook: *Separation Process Engineering* [1]. During these lectures, the celebrated Rayleigh's equation is derived. Then, simple integration of this equation using graphical or numerical (Simpson's rule) techniques is used to find, for example, the final still pot holdup, given the final composition in the still pot, the initial feed holdup and composition, and the vapor–liquid equilibrium (VLE) data. Usually, students taking CHE 409 have 2 weeks to use the experimental data obtained during the laboratory session in order to submit a report, in which some chemical engineering calculations must be included. It turns out, as this article will describe in detail, that several interesting calculations can be made in the case of a binary batch distillation. We believe that this new experiment and its related computations help bridge the gap between the theoretical lectures of the CHE 306 course and the experimental work

of the CHE 409 laboratory. In addition, by solving both mass and energy balances in order to describe the transient behavior of a simple batch distillation, the authors go beyond what is usually covered in CHE 306 at KFUPM or equivalent junior courses at other institutions. This article contains five sections.

1. The authors start by showing how vapor–liquid equilibrium data, for the nonideal binary mixture composed of ethanol and water, can be computed using both the modified Raoult's law and either the Wilson, NRTL, van Laar, Margules, or UNIQUAC models for activity-coefficient predictions.
2. The apparatus for simple batch distillation is then summarily described.
3. The governing mass and energy equations that allow the computation of the transient behavior of the still pot temperature and composition are described next.
4. The next section gathers all experimental data together with the theoretical predictions, and these results are thoroughly discussed.
5. Finally, the authors conclude by sharing with the readers of this article other ideas and experiments that can be pursued using the same experimental apparatus or more involved ones.

A recent graduate level textbook discusses advanced batch distillation problems in depth [2]. A literature survey shows that only few articles have tackled the simple batch distillation problem from an educational perspective. A multiBatch distillation system for simulation, optimal design, and control of batch distillation has been presented by Diwekar [3]. A virtual

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laboratory experiment on batch rectification was built at the Johns Hopkins University by Fleming and Paulaitis [4]. Liu and Peng [5] used Microsoft Excel to simulate the simple batch distillation of a multicomponent mixture. Silva et al. [6] have used an Oldershaw column to conduct experimental batch rectification. None of the above-mentioned articles has attempted to combine both experimental and theoretical considerations to study simple batch distillation.

In the present work, Mathematica® was used extensively to predict the theoretical transient behavior of the simple batch experiment. The computer code can be obtained from the corresponding author upon request.

VLE DATA FOR THE ETHANOL–WATER MIXTURE

The binary mixture composed of ethanol and water present a pressure-sensitive positive azeotrope. The boiling temperature and composition of the azeotrope are found to be equal to 78.2°C and 89 mol% ethanol if the pressure is 101.325 kPa. The VLE data (i.e., equilibrium curve and isobaric VLE diagram) at low to moderate pressures can be readily obtained using the modified Raoult's law and the Wilson's model [7] for activity coefficient prediction. The modified Raoult's law can be written as follows:

$$y_i P = x_i P_i^{\text{sat}} \gamma_i, \tag{1}$$

where P_i^{sat} is the vapor pressure and γ_i the activity coefficient of component i, which can be obtained from

$$\ln(\gamma_k) = -\ln\left(\sum_{j=1}^{N_c} x_j A_{kj}\right) + 1 - \sum_{i=1}^{N_c} \frac{x_i A_{ik}}{\sum_{j=1}^{N_c} x_j A_{ij}}, \tag{2}$$

In Equation (2) (i.e., the Wilson model), A_{ij} is the binary interaction parameter, which depends on the molar volumes (v_i and v_j) and the energy terms (λ_{ij} and λ_{ii}),

$$A_{ij} = \frac{v_j}{v_i} \exp\left(-\frac{\lambda_{ij} - \lambda_{ii}}{RT}\right). \tag{3}$$

Using Mathematica® the authors have obtained such VLE data for different values of the pressure, which can be set by the slider. Various activity coefficient models (e.g., van Laar, Margules, NRTL, UNIQUAC, and Wilson) have been considered as can be seen in Figure 1.

SIMPLE BATCH DISTILLATION EXPERIMENTAL SET-UP

A rudimentary experimental apparatus is sufficient to get good data quickly. The set-up, sketched in Figure 2, is composed of a still pot heated with an electrical wire, two thermocouples with digital display, a condenser, and a distillate receiver. Both apparatus cost, complexity, and size are insignificant if compared to our continuous distillation column of the CHE 409 laboratory, which requires a dedicated technician to be operated. The experimental apparatus is used at atmospheric pressure (i.e., 101.325 kPa). For more accurate data collection, authors advise readers to use a manometer in order to have a precise measurement of pressure since the collected data (i.e., the still pot and distillate temperatures) are sensitive to the value of the operating pressure. Also, the VLE data vary significantly with pressure. For instance, if the pressure is 95 kPa, the boiling temperature of water will be quite different from the normal boiling temperature of water, 100°C.
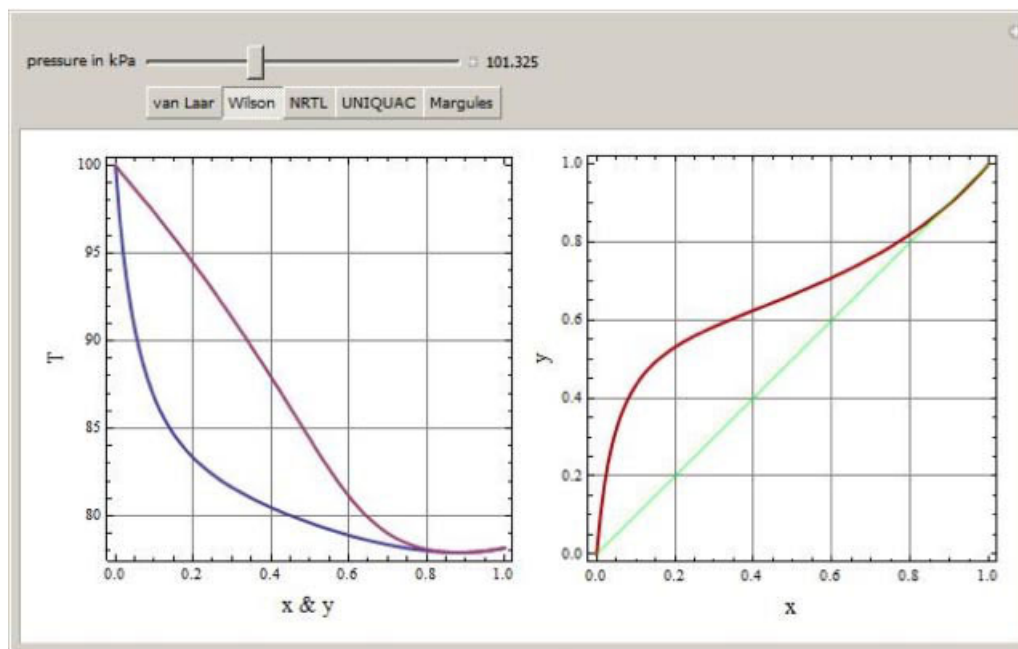
Figure 1   Computed VLE data for the ethanol–water binary mixture at $P = 101.325$ kPa. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

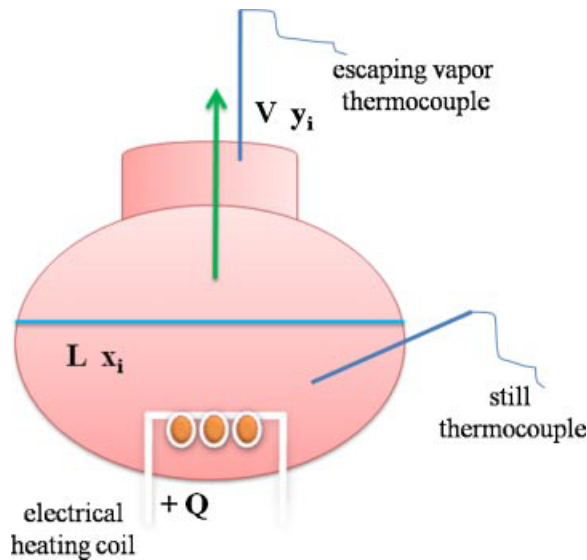**Figure 2** Experimental set-up sketch. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

MASS AND ENERGY BALANCE EQUATIONS

A simple batch distillation experiment consists in two stages: (1) heating the mixture in the still pot until it reaches its boiling temperature and (2) conducting the actual distillation experiment. Both periods are rich in information and can be modeled theoretically.

Heating Period

In the heating period sensible heat is added to the mixture using a heating coil at a rate equal to $\dot{q}$ (expressed in kW). The governing equation is as follows:

$$n\, C_\mathrm{P}(T)\frac{\mathrm{d}T}{\mathrm{d}t} = \dot{q}, \qquad (4)$$

where $n = M(0)$ is the total number of moles initially fed to the still pot, T is the temperature, and $C_\mathrm{P}(T)$ is the mixture heat capacity expressed kJ/(kmol K). Since the dependency of C_P versus temperature is small in our case study (i.e., the particular ethanol–water binary mixture) and $\dot{q}$ is constant (i.e., we are assuming a constant-heating policy during both heating and distillation periods), $T(t)$ is approximately a linear function of time.

Distillation Phase

A global and component mass balances around the still pot are given by the following equations:

$$\frac{\mathrm{d}M}{\mathrm{d}t} = -V, \qquad (5)$$

$$\frac{\mathrm{d}(Mx)}{\mathrm{d}t} = -Vy, \qquad (6)$$

where x is the ethanol mole fraction in the still pot, M is the molar holdup in the still pot, V is the escaping vapor molar flow rate, and y is the vapor mole fraction of ethanol.

The energy balance equation written around the still pot, which requires a good grasp of enthalpy calculations by chemical engineering students, is given by

$$\frac{\mathrm{d}(Mh)}{\mathrm{d}t} = -VH + \dot{q}, \qquad (7)$$

where H is the molar enthalpy of the escaping vapor stream and h is the molar enthalpy of the liquid in the still pot. Solution of the governing equations requires good VLE data and the built-in Mathematica® function, NDSolve, which allow the integration of the above differential-algebraic system of equations. Indeed, in addition to the differential equations (i.e., Eqs. 5–7), one has to use the following equilibrium algebraic equation: $y = y_\mathrm{eq}(x)$, which reflects that the escaping vapor is in equilibrium with the liquid in the still pot. It is worth listing the assumptions behind Equation (7): (1) the holdup in the vapor phase is negligible and (2) the molar enthalpy and molar internal energy of the liquid phase are equal.

EXPERIMENTAL DATA AND THEORETICAL PREDICTIONS

A mixture composed of 50 mol% ethanol and 50 mol% water was used in the simple batch distillation experiment at $P = 101.325$ kPa. A constant heating policy was adopted. Experimental data along with the theoretical prediction for the heating phase are given in Figure 3. The theoretical curve is shown in red and the value of $\dot{q}$ was adjusted so that the theoretical predictions fit well the experimental data (i.e., temperature in the still pot versus time). The initial molar holdup in the still pot was 23.4 mol and the duration of the heating period was around 1,450 s. An approximate value of $\dot{q}$ equal to 85.5 W was found. This value is quite close to the rated power of the electrical resistance used, which is equal to 110 W. This
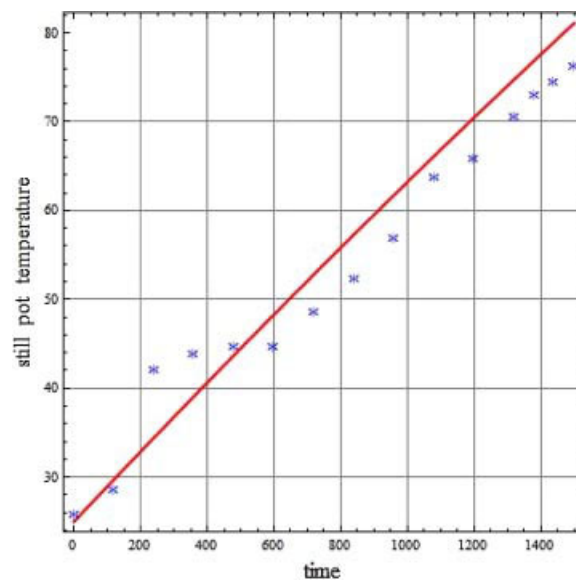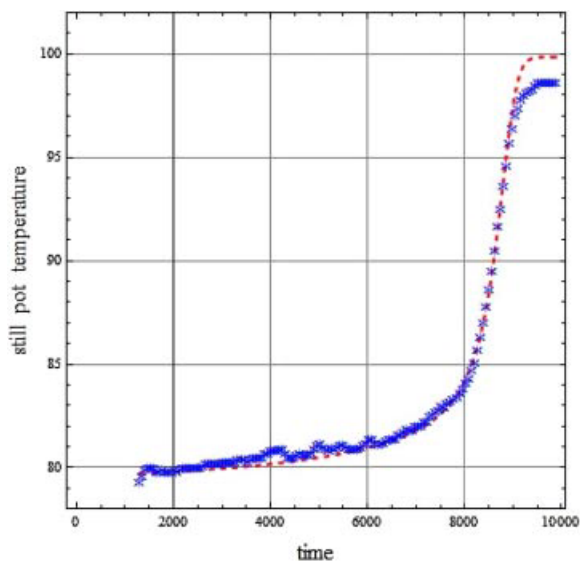

Figure 3 $T_\mathrm{still}(t)$ (expressed in °C) versus time in seconds during the heating period (the red line is the theoretical prediction). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
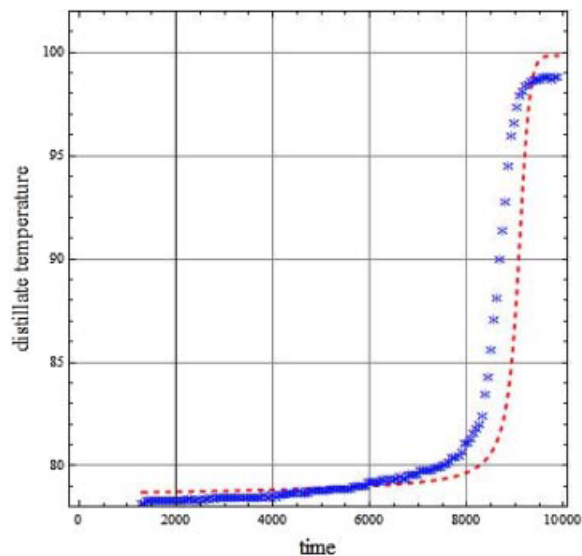
is particularly reassuring since no heat losses were accounted for in our model. The final value of the temperature is around 80°C, which corresponds to the boiling point of the equimolar ethanol–water mixture initially fed in the still pot.

When the distillation phase started both temperatures, of the still and the vapor escaping the still, versus time were recorded every minute. The experimental data and the theoretical predictions show only qualitative agreement and are presented in Figures 4 and 5. Observed discrepancies can be accounted for by noting the still pot was not insulated and the molar enthalpy expressions used in the theoretical model did not involve temperature-dependent gas and liquid heat capacities and temperature-dependent latent molar heats. In addition, we are assuming that a cooling system (not shown in Fig. 2) is properly adjusted to obtain a saturated distillate liquid stream. The composition of this stream is given by $x_{\text{distillate stream}} = y_{\text{leaving vapor}}$. Then, the theoretical distillate temperature is just the boiling temperature of this distillate stream. On the other hand the experimentally measured temperature corresponds to the temperature of the escaping vapor measured using a thermocouple placed at the entrance of the condenser of our experimental apparatus.

If one plots the still and distillate temperatures versus time in the same plot, one can see that as expected the distillate temperature lags behind the still pot temperature (see Fig. 6). This is because the still pot gets depleted in the light component while the distillate is enriched in ethanol. Finally, only pure water remains in the still pot at the end of the distillation run and the temperature is equal to 100°C. This distillation phase lasted from $t_1 = 1,450$ s to $t_2 = 10,050$ s. If one assumes that the vapor flow rate, V, leaving the still pot is constant, then energy balance equation is not needed anymore and good enough results can be obtained as one can see in Figure 7. V may be easily computed from: $M(t = 0) = V(t_2 - t_1)$. For our experiment, we have found V equal to 2.73×10^{-3} mol/s.
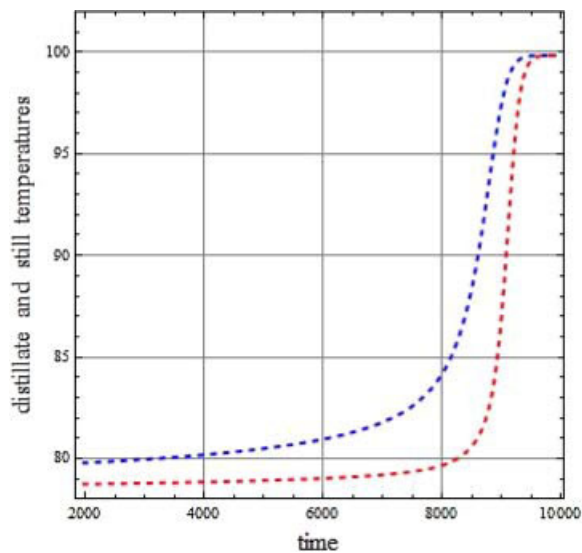

Figure 5 $T_{\text{distillate}}(t)$ (expressed in °C) versus time in seconds during the distillation phase (Experimental data and theoretical prediction are shown using blue * and dashed red curve, respectively). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
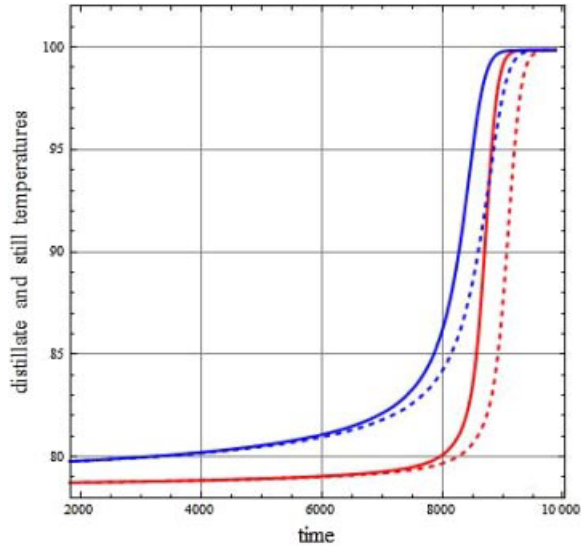
An interesting exercise for the students is to learn to infer compositions from experimental temperature measurements using the following equation: $P = \sum_{i=1}^{2} x_i P_i^{\text{sat}} \gamma_i$. It is important to note that this is possible only for binary mixtures. The authors used the still pot temperature measurements to compute the composition in the still pot versus time. These "experimental" or inferred compositions can be compared to the result obtained using our rigorous simulation with mass and energy

Figure 4 $T_{\text{still}}(t)$ (expressed in °C) versus time in seconds during the distillation phase (Experimental data and theoretical prediction are shown using blue * and dashed red curve, respectively). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
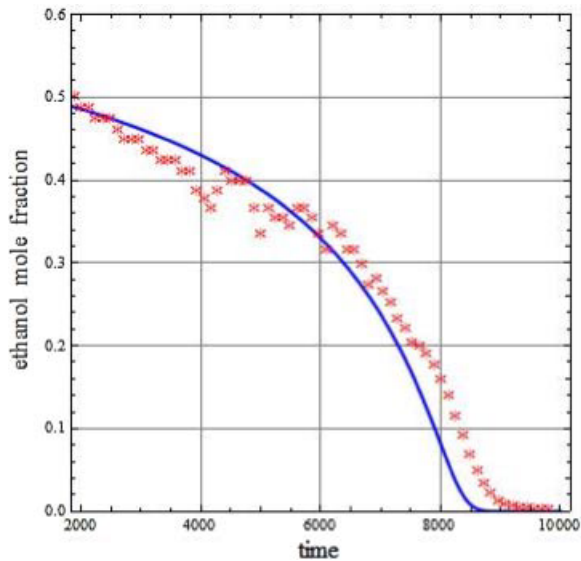

Figure 6 $T_{\text{distillate}}(t)$ and $T_{\text{still}}(t)$ (both expressed in °C) versus time in seconds during the distillation phase (blue color corresponds to $T_{\text{still}}(t)$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

dimensionless warped time, ξ, as follows:

$$\xi = \frac{1}{a-1+b}\ln\left(\frac{x_0(1-x)}{x(1-x_0)}\right) + \frac{(a-1)^2}{(a-1+b)(a-1+ab)}$$
$$\ln\left(\frac{(1-x)(a-1+b[1+(a-1)x_0])}{(1-x_0)(a-1+b[1+(a-1)x])}\right), \tag{8}$$
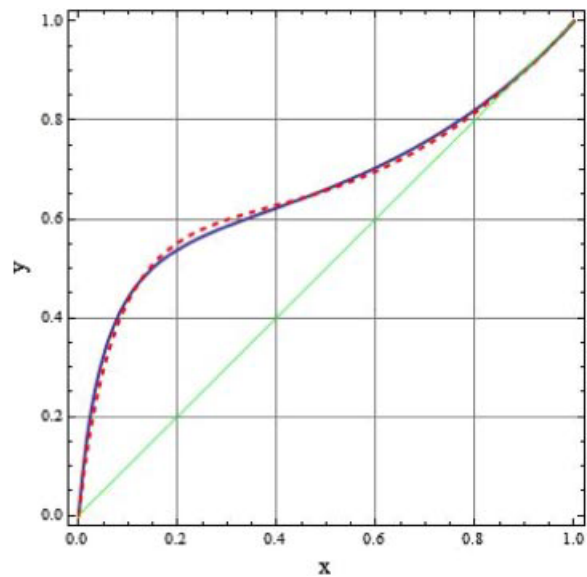
where x_0 is here equal to 0.5 (i.e., the feed composition) and a and b parameters to be determined so that the VLE data for ethanol–water mixture can be represented by the equation below:

$$y = \frac{ax}{1+(a-1)x} + bx(1-x), \tag{9}$$

A simple least-squares analysis using the VLE data computed with the Wilson model gives $a = 9.88722$ and $b = -1.0023$. Reader must be cautious when using Equation (9) since it is strictly valid for binary mixtures with a minimum-boiling azeotrope such as ethanol–water or ethanol-ethylacetate... but not valid for mixtures such as acetone-chloroform, which present a maximum-boiling azeotrope. The VLE data obtained with Equation (9) agree well with the rigorous VLE data obtained using the Wilson model and the modified Raoult's law as can be seen in Figure 9.

Since the dimensionless warped time concept, first introduced by Doherty and Perkin [9], is difficult to grasp (i.e., ξ is a nonlinear function of time given by $d\xi = (V/(M(t)))dt$ where $M(t)$ is the time-dependent molar holdup in the still), we have intentionally chosen to give the expression of $\xi(t)$ for our case study and to plot this function of clock time (see Eq. 10 and Fig. 10).

$$\xi(t) = 3.1355 - \ln(26.5511 - 0.002731t) \tag{10}$$

Figure 7 $T_{distillate}(t)$ and $T_{still}(t)$ (both expressed in °C) versus time in seconds during the distillation phase (theoretical prediction with and without energy balance are shown using dashed and solid curves, respectively; blue color corresponds to $T_{still}(t)$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

balances. Such comparison is shown in Figure 8. We see that the composition in the still pot (i.e., ethanol mole fraction) drops from 50 mol% ethanol (i.e., the feed composition) to 0 mol% ethanol or pure water at the end of the experiment. A theoretical derivation made by Doherty and Malone [8] shows that the composition in the still pot depends implicitly on the

Figure 8 Ethanol mole fraction in the still versus time (theoretically computed composition is shown in solid blue curve and temperature-inferred composition is shown using red *). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 9 Ethanol–water VLE data at $P = 101.325$ kPa (using the rigorous approach in solid blue and using Eq. 9 in dashed red). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
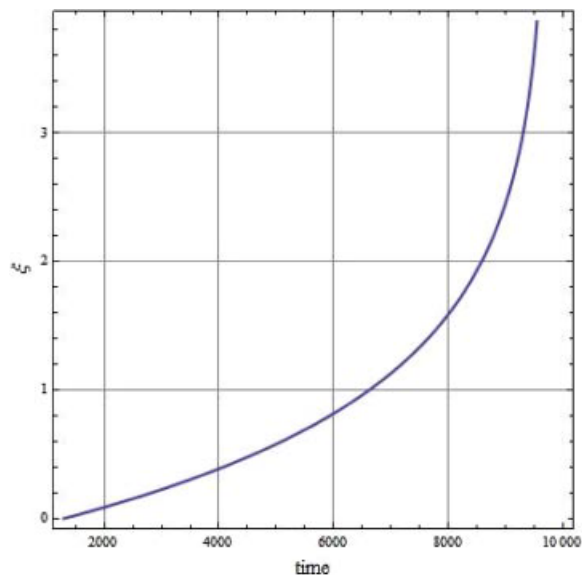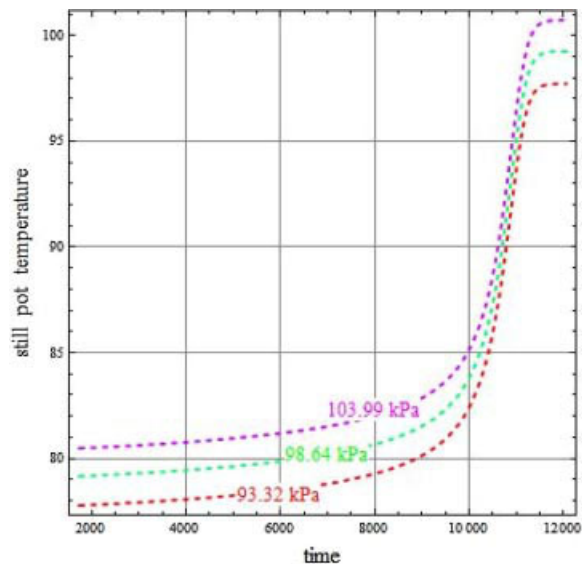
Figure 10 Dimensionless warped time versus clock time. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 11 Effect of pressure on the behavior of T_{still} versus time. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Equation (10) can be used in conjunction with Equation (8) to get the theoretical prediction of the still's composition versus clock time shown in Figure 8.

PERSPECTIVES OF PRESENT WORK

Several potential extensions of the present work are suggested to the readers.

1. It is possible to explore the effect of using a pressure other than 101.325 kPa both numerically and experimentally. Here we show (Fig. 11), what to expect by setting P to 93.32 and 103.99 kPa
2. Another venue, which the readers might want to explore, is the use of batch rectification instead of simple batch distillation. Of course, the equipment now is more complex and the manipulation is more involved. A preliminary calculation shows what one would get using several columns with varying number of stages (e.g., 2, 6, and 15 stages). Reflux ratio was kept constant equal to 1.5 throughout all numerical computations, which used constant molal overflow (CMO) assumption. Results, shown in Figure 12, indicate that the separation is enhanced as the number of stages increases. As the number of stages is increased, one gets a distillate composition close to the azeotropic composition (i.e., 89 mol% ethanol at $P = 101.325$ kPa). Initially, the column has the same composition as the mixture fed to the still (i.e., equimolar binary mixture composed of ethanol and water). This explains the observed slight decrease in the distillate temperature at small times.
3. If one takes the acetone–chloroform binary mixture, which exhibits a negative azeotrope at atmospheric pressure, then he can run a similar simple batch experiment.

The corresponding results would lead to different interpretations and show other fundamental aspects of distillation. Indeed, at the end of the distillation run the residue in the still pot will have exactly the azeotropic composition and temperature (33.72 mol% acetone and 64.53°C at $P = 101.325$ kPa, respectively). The residue can be collected and analyzed using gas-chromatography (GC) in order to get experimental values for the azeotropic composition.
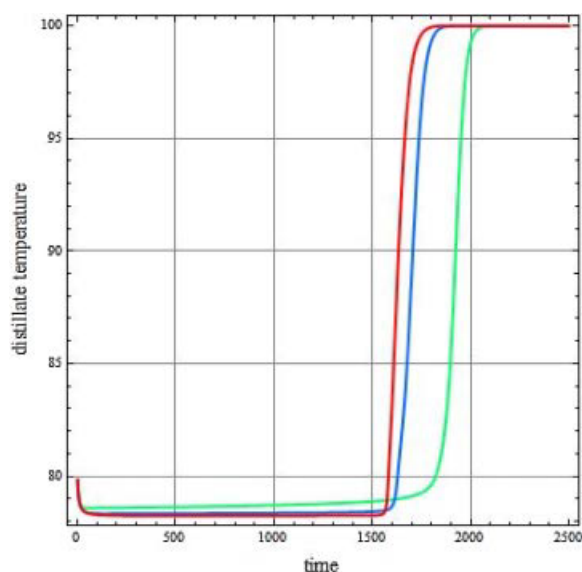

Figure 12 Batch rectification with two-stage (green curve), six-stage (blue curve) and fifteen-stage (red curve) columns. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

4. One can separate *n*-octane from *n*-dodecane using the same apparatus if he adds water to this mixture of the two normal paraffinic hydrocarbons. The experiment falls under the category called multicomponent steam distillation since water is added. A theoretical treatment of this problem is described in detail by Ingham et al. [10]. Figure 13 shows what one would get using Mathematica$^®$ when the distillation is conducted at 101.325 kPa. It is worth noting that the separation will take place below the normal boiling point of water (i.e., 100°C). Indeed, one would expect the *n*-octane to exit the still pot in the form of a mixture with water (i.e., a minimum-boiling heteroazeotrope with temperature around 93°C). At a later stage, *n*-dodecane will also escape the still pot accompanied with water in the form of a minimum-boiling heteroazeotrope with temperature around 99°C.

5. Finally, one could feed a ternary mixture composed of 20 mol% ethanol, 20 mol% *n*-propanol and 60 mol% *n*-butanol feed (or any other constant relative volatility mixture) to the simple batch distillation apparatus. If a run is conducted at atmospheric pressure, one can collect samples from the still pot at various times and analyze them with GC. Experimental data and computed residue curve obtained with Mathematica$^®$ at $P = 101.325$ kPa

(see Fig. 14) should be consistent. This introduces students to another aspect of distillation called conceptual design and using residue curve maps (RCMs). More advanced labs could involve other ternary systems such as ethanol–water–ethylene glycol or acetone–chloroform–methanol, which exhibit deviation from ideal behavior and require the use of the Modified Raoult's law and activity prediction models such as the Wilson model.

CONCLUSION

Several experimental and numerical results concerning the batch distillation of an ethanol–water mixture were presented. These results are easy enough to obtain and analyze that they seem suitable to a senior laboratory course on unit operations. Other interesting and more involved experiments were proposed in the last section of this article. They include batch rectification and RCM determination. It is our belief that the proposed experiment and related calculations can strengthen the understanding of undergraduate student of three different topics: distillation, numerical computations in chemical engineering, and chemical engineering thermodynamics.
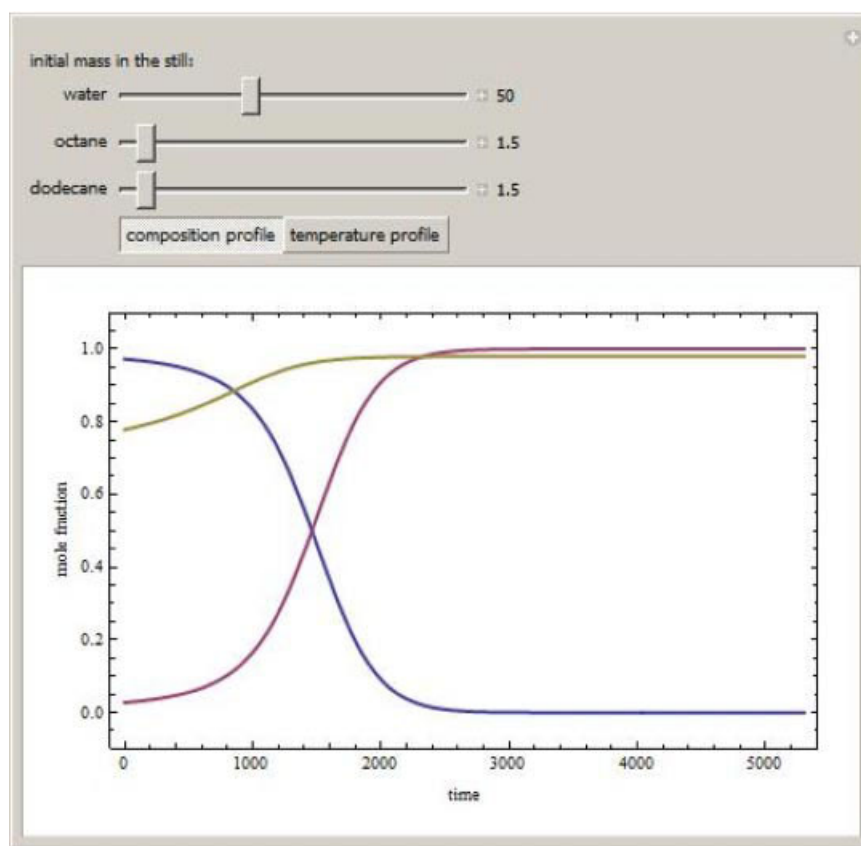
Figure 13 Separation of *n*-octane and *n*-dodecane at 101.325 kPa using steam distillation (mole fractions of *n*-octane and *n*-dodecane in the distillate on a water-free basis are shown in solid blue and solid magenta; water mole fraction in the distillate is shown in solid brown). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]
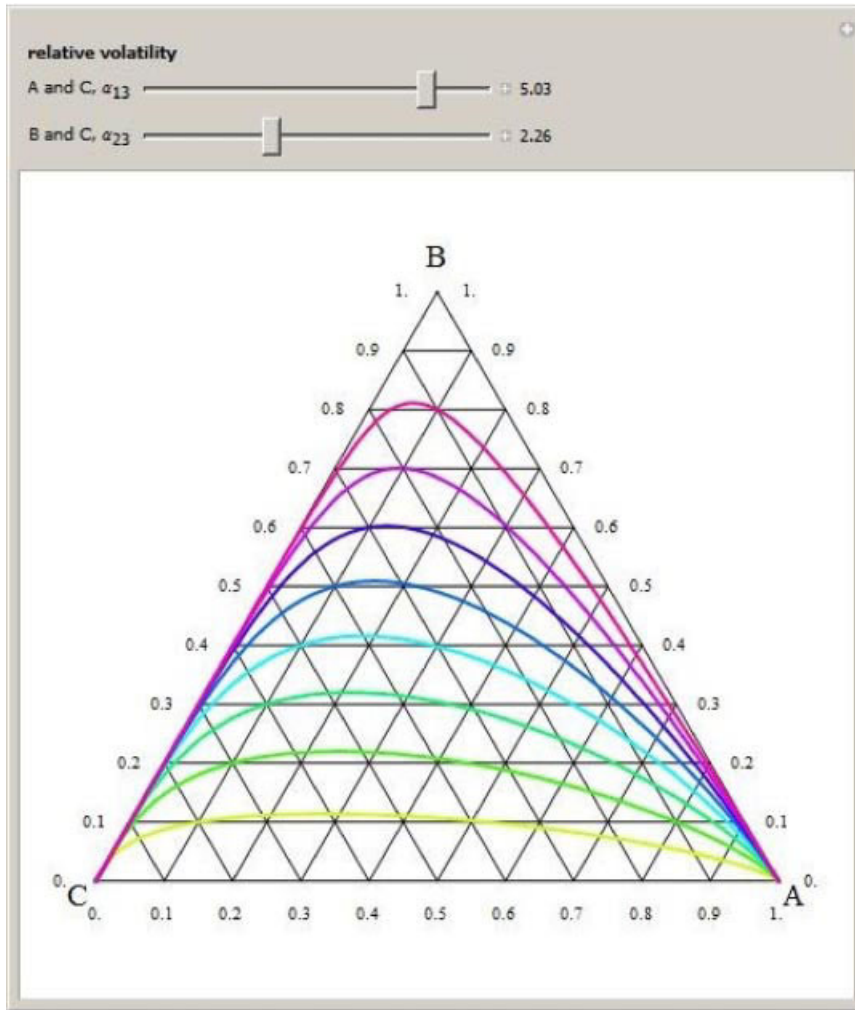
Figure 14 Residue curve map (RCM) for a constant relative volatility mixture such as ethanol–*n*-propanol–*n*-butanol (EPB) mixture (slider's value should read: $\alpha_{EB} = 5.03$ and $\alpha_{PB} = 2.26$). [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

ACKNOWLEDGMENTS

NOMENCLATURE

C_P	heat capacity (J/mol K)
h	liquid enthalpy (J/mol)
H	escaping vapor molar enthalpy (J/mol)
M	molar holdup of the still pot (mol)
P	total pressure (kPa)
P_i^{sat}	vapor pressure (kPa)
$\dot{q}$	heating power (kW)
t	clock time (seconds)
T	temperature (°C)
v	molar volume (cm³/mol)

V	molar flow rate of escaping vapor (mol/s)
x	liquid mole fraction
y	vapor mole fraction

Greek Symbols

α	relative volatility
λ	energy term (kcal/kmol)
γ	activity coefficient
ξ	dimensionless warped time

APPENDIX

The Antoine constants for vapor pressure calculations and the parameters for the Wilson model for the ethanol and water mixture are given in Tables A1 and A2:

Table A1 Antoine's Constants for Ethanol and Water With $\log_{10}(P^{\text{sat}}(\text{mmHg})) = A - (B/(C + t(^{\circ}C)))$

	A	B	C
Ethanol	7.68117	1332.04	199.200
Water	8.07131	1730.63	233.426

Table A2 Wilson Model Parameter for Ethanol and Water Mixture

λ_{ij}	Ethanol	Water	Molar volume
Ethanol	—	975.4859	58.49
Water	276.7557	—	17.88

Table A3 Heat Capacity Data and Latent Heats for Ethanol and Water

	$C_{\text{P},i}^{\text{L}}$ (J/mol K)	$C_{\text{P},i}^{\text{V}}$ (J/mol K)	λ_i (J/mol)
Ethanol	163.80	73.98	38657.86
Water	75.66	38.29	41780.00

Liquid and vapor phase enthalpies are calculated using the following simplified equations [1], which assume ideal gas-phase and ideal solution behavior:

$$h_{\text{L}}(x, T) = x_1 C_{\text{P},1}^{\text{L}}(T - T_{\text{ref}}) + x_2 C_{\text{P},2}^{\text{L}}(T - T_{\text{ref}}) \tag{A.1}$$

and

$$H_{\text{V}}(y, T) = y_1[\lambda_1 + C_{\text{P},1}^{\text{V}}(T - T_{\text{ref}})]$$
$$+ y_2[\lambda_2 + C_{\text{P},2}^{\text{V}}(T - T_{\text{ref}})] \tag{A.2}$$

where the heat capacities, $C_{\text{P},i}^{\text{L}}$ and $C_{\text{P},i}^{\text{V}}$, and the latent heat, λ_i at $T_{\text{ref}} = 78.17^{\circ}C$, of component $i = 1$ or 2, are given in Table A3.

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BIOGRAPHIES

Dr. Housam Binous, a visiting Associate Professor at King Fahd University Petroleum & Minerals, has been a full time faculty member at the National Institute of Applied Sciences and Technology in Tunis for eleven years. He earned a Diplôme d'ingénieur in biotechnology from the Ecole des Mines de Paris and a PhD in chemical engineering from the University of California at Davis. His research interests include the applications of computers in chemical engineering.

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