

# CONTINUOUS AND BATCH DISTILLATION IN AN OLDERSHAW TRAY COLUMN

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**D**istillation is by far the most frequently used industrial separation process. Although not energy-efficient, it has a simple flowsheet and is a low-risk process. It is indeed the benchmark with which all newer competitive processes must be compared. Following Null,<sup>[1]</sup> distillation should be selected if the relative volatility is greater than 1.05, whereas Nath and Motard<sup>[2]</sup> and Douglas<sup>[3]</sup> indicate  $\alpha_{12}$  greater than 1.10, a more conservative critical value for the relative volatility. Generally, design heuristics point out that processes using energy separation agents should be favored.

For the reasons outlined above, distillation experiments are included in the Chemical Engineering Integrated Master curriculum of the Department of Chemistry at University of Aveiro (DCUA). Students start receiving lectures on distillation as part of the Separation Processes I course, which is essentially devoted to equilibrium-staged unit operations. Afterwards, experiments are carried out in Laboratórios EQ (Chemical Engineering Laboratory), a weekly six-hour lab course intended to provide hands-on experience on separations, reaction, and control. Each experiment lasts two weeks: in the first week students—divided into groups of three—carry out the lab exercise and some calculations, and in the second week students do numerical calculations and computer simulations, which require computational support. Student assessment is based on a very short individual oral quiz and a report prepared by the student groups.

In this paper a lab exercise on continuous and batch rectification developed at DCUA is presented. Papers with experimental work in the distillation field are scarce and accordingly this communication intends to fill this gap. There are a number of educational publications concerning distillation

calculations, mostly using Excel, Matlab, Hysys, and Mathematica software.<sup>[4-6]</sup> Moreover, virtual laboratories involving distillation units have been developed in order to enhance the understanding of the process units and to improve the teaching effectiveness.<sup>[7, 8]</sup> Nonetheless, students are usually uninterested in a problem unless they can visualize it in practice, so experiments in the lab should never be totally replaced by simulated experiments on a computer, notwithstanding its ease and less time-consuming approach.

In this work, experiments are performed in an Oldershaw column with five sieve trays to separate cyclohexane/n-heptane under different modes of operation. These modes include total reflux, continuous rectification with partial reflux, and

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batch rectification with constant reflux. An Oldershaw tray column is a laboratory-scale column equipped with perforated trays. Of special importance is the fact that it exhibits a separation capacity close to that of large industrial columns.<sup>[9]</sup> In fact, experimental results show that commercial towers will require a similar number of stages to reach the same separation level obtained in the Oldershaw unit.<sup>[10]</sup>

With this work students practice relevant concepts introduced earlier in their curriculum, namely vapor-liquid equilibrium, continuous vs. batch operation, McCabe-Thiele graphical method, column efficiency, and application of the generalized Rayleigh equation. Moreover, students use industrial simulation software (Aspen) to predict experimental results, giving them the opportunity to improve their skills in this field, too. By examining experimental results and comparing them with those obtained from simulations, students gain insight to this unit operation.

## LABORATORY DESCRIPTION

### Experimental Setup

Experiments are performed in an Oldershaw tray column instrumented and equipped with a control system supplied by Normschliff Gerätebau (similar equipment is available from Normag GmbH Imenau). Other commercial teaching equipment for continuous distillation is offered, for example, by Armfield, Ltd. (<[www.discoverarmfield.co.uk](http://www.discoverarmfield.co.uk)>), De Dietrich-QVF (<[www.ddpsinc.com](http://www.ddpsinc.com)>), and Phywe (<[www.phywe-systeme.com](http://www.phywe-systeme.com)>). The unit used is shown in Figure 1 and comprises five perforated plates (3 cm of diameter), a reboiler

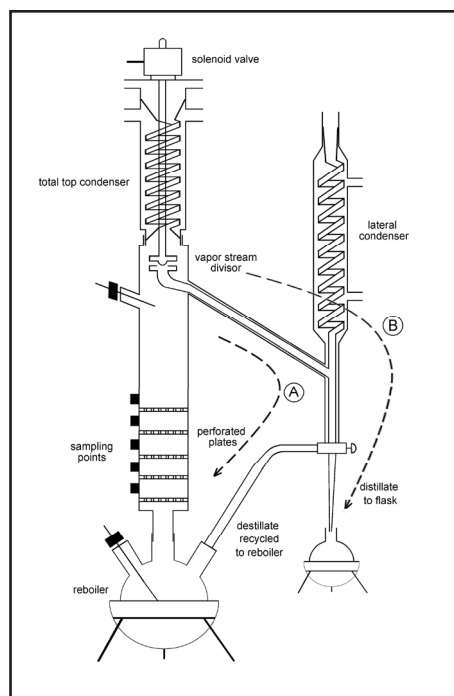


Figure 1. Oldershaw tray column.

(capacity of 2 L), a total top condenser using tap water as cooling fluid, a lateral condenser to remove distillate as liquid, and a solenoid valve to divide the vapor stream into reflux and distillate under the partial reflux mode. Additional features include: sampling points above each tray to determine liquid composition; and tempera-

ture sensors immersed in the reboiler and located in the top condenser allowing the determination of the bottom and head compositions, respectively. The column is used to separate c.a. 800 mL of a cyclohexane (Lab-Scan, 99%) / n-heptane (Lab-Scan, 99%) mixture with 30% (mol) of cyclohexane.

The calibration curve—measured in this work—to determine the cyclohexane mole fraction ( $x_1$ ) in a cyclohexane–n-heptane mixture at 30 °C as function of refractive index (RI), is given by  $x_1 = -309.95 \text{ RI}^2 + 895.15 \text{ RI} - 645.15$ .

### Experiments at Total Reflux, $R = \infty$

Rectifications at total reflux were performed at two distinct effective reboiler powers ( $P = 75$  and  $125$  W) to evaluate the effect of the internal molar flow upon separation and column efficiency. The invariance of the top and bottom ( $T_D$  and  $T_B$ ) temperatures was used to detect the steady state. Additionally, they were utilized to determine the corresponding cyclohexane molar compositions,  $x_D$  and  $x_B$ , by vapor-liquid equilibrium calculations assuming that the column is kept at atmospheric pressure (pressure drop along the column is considered negligible).

### Continuous Rectification at Partial Reflux

This Oldershaw tray column is extremely versatile. It can be operated continuously under partial reflux. With simple modifications, the distillate may be directly fed to the reboiler (see path A in Figure 1), allowing us to reach the corresponding steady state. Such an experiment was carried out at  $R = 6$  for  $P = 125$  W. Once more,  $T_D$  and  $T_B$  were utilized to determine  $x_D$  and  $x_B$ .

### Batch Rectification at Constant Partial Reflux

Finally, a semi-continuous or batch distillation was performed for  $R = 6$  and  $P = 125$  W. Presently, the distillate is not fed to the reboiler, but collected in the independent flask shown in Figure 1 (see path B). Under such mode of operation, compositions vary along time.  $T_D$  and  $T_B$  were registered during 1 h approximately, to calculate the corresponding  $x_D$  and  $x_B$ , and the distillate refractive index was measured at the end.

## HAZARDS AND SAFETY PRECAUTIONS

Cyclohexane (CAS registry number: 110-82-7) and n-heptane (CAS registry number: 142-82-5) are stable liquids at room temperature, highly flammable, and may readily form explosive mixtures with air. They are harmful if swallowed or inhaled, and cause irritation to skin, eyes, and respiratory tract. Attention must be paid during the withdrawal of liquid samples, from the bottom of the column, in order to measure the refractive index. Protection equipment, including gloves and glasses, should be used. Students must review the Materials Safety Data Sheet for each chemical before starting the experiment and are instructed to collect wastes in specific tanks to be subsequently treated by the DCUA.

## DATA ANALYSIS

### Vapor-Liquid Equilibrium

At low pressure, vapor-liquid equilibrium of a component  $i$  may be represented by:

$$y_i P_t = x_i \gamma_i(\underline{x}) P_i^\sigma(T) \quad (1)$$

where  $y_i$  and  $x_i$  are the vapor and liquid molar fractions, respectively,  $P_i^\sigma$  is its vapor pressure,  $\gamma_i$  is its activity coefficient, and  $P_t$  is total pressure.  $P_i^\sigma$  is computed by the Antoine equation and  $\gamma_i$  by Margules equations, whose constants may be found in the literature.

Since  $\sum x_i = \sum y_i = 1$ , the liquid molar fraction may be determined for any temperature by the relation:

$$P_t = x_1 \gamma_1(\underline{x}) P_1^\sigma(T) + (1 - x_1) \gamma_2(\underline{x}) P_2^\sigma(T) \quad (2)$$

where  $\underline{x}$  denotes the liquid composition vector. The vapor molar fraction can be then determined by Eq. (1).

### Number of Equilibrium Stages

The number of equilibrium stages is obtained by the well-known McCabe-Thiele method.<sup>[11]</sup> In this work the column has a rectifying section only, hence the operating line is:

$$y_{n+1} = \left( \frac{R}{R+1} \right) x_n + \left( \frac{1}{R+1} \right) x_D \quad (3)$$

where  $y_{n+1}$  and  $x_n$  are the cyclohexane vapor and liquid fractions of trays  $n+1$  and  $n$ , respectively. At total reflux ( $R = \infty$ ) the operating line coincides with the diagonal line. The number of equilibrium stages is given by the number of outlined steps between  $x_D$  and  $x_B$ . The number of trays is obtained by

subtracting one stage (corresponding to reboiler) from the total number of equilibrium stages.

### Overall Efficiencies

The experimental overall efficiency is given by:

$$E_{ov}(\%) = \frac{N_{ideal}}{N_{real}} \times 100 \quad (4)$$

where  $N_{ideal}$  is the ideal number of equilibrium stages and  $N_{real}$  is the actual number of trays (in this case  $N_{real} = 5$ ).

The overall efficiency can be estimated by empirical correlations, namely, those by Drickamer and Bradford<sup>[12]</sup> and O'Connell.<sup>[13]</sup> Drickamer and Bradford<sup>[12]</sup> correlate  $E_{ov}$  with the feed viscosity,  $\mu$ , at the average temperature of the column:

$$E_{ov}(\%) = 13.3 - 66.8 \log \mu(\text{cP}) \quad (5)$$

O'Connell used a viscosity and relative volatility,  $\alpha_{12}$ , dependence. His graphical result can be fit with

$$E_{ov}(\%) = 50.3 \left[ \alpha_{12} \times \mu(\text{cP}) \right]^{-0.226} \quad (6)$$

where  $\alpha_{12}$  is the geometric average of the bottom and top values.

### Generalized Rayleigh Equation

The moles of liquid in the reboiler are related to its residue composition by the Generalized Rayleigh equation:

$$\ln \frac{B}{F} = \int_{x_{B,0}}^{x_{B,final}} \frac{dx_B}{x_D - x_B} \quad (7)$$

where  $F$  and  $B$  are the initial and final moles of mixture in the reboiler, respectively. Knowing experimental pairs of data ( $x_D$ ,  $x_B$ ),  $B/F$  fraction may be obtained by numerical integration.

### Results and Discussion

In Table 1 the results obtained at total reflux at 75 and 125 W are presented. For illustration, the McCabe-Thiele diagram for 125 W is plotted in

**Figure 2.** McCabe-Thiele diagram for a) total reflux distillation and b) continuous rectification at partial reflux.

**TABLE 1**  
Experimental Conditions and Results for the Experiments at Total Reflux

P(W)	$T_D(^{\circ}\text{C})$	$T_B(^{\circ}\text{C})$	$x_D$	$x_B$	$E_{ov}(\%)$		
					Exp.	Eq. 5	Eq. 6
75	83.9	92.6	0.878	0.281	95.1	53.1	64.8
125	84.2	92.7	0.864	0.273	92.1	53.2	64.9

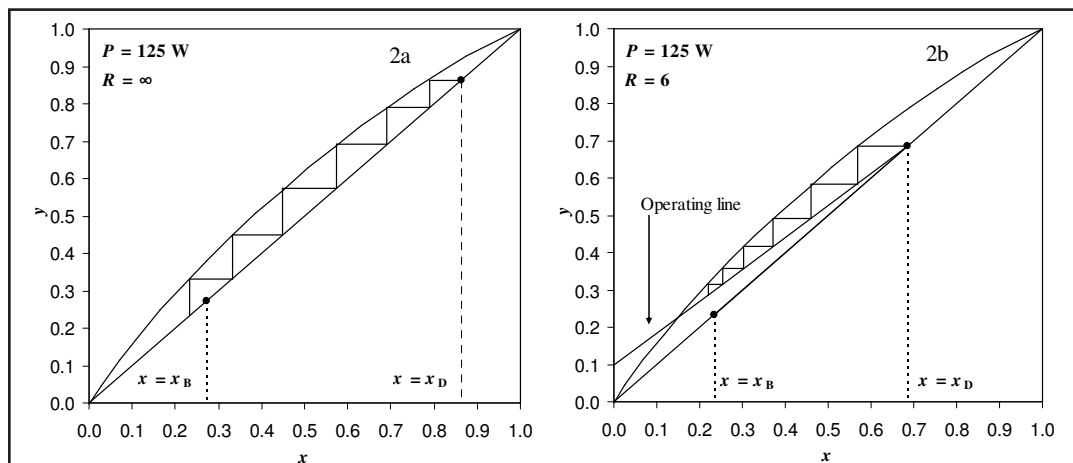


Figure 2a. The minimum number of equilibrium stages was 4.76 and 4.61 for  $P = 75$  and  $125$  W, respectively, giving rise to overall efficiencies of 95.1% and 92.1%. These results indicate the column is more efficient when operated at 75 W, which is usually unexpected for the students. Actually, higher reboiler powers generate higher internal flows. Although such effect may lead to a foreseen increase of mass transfer coefficients, it also decreases the mean residence times of both phases in each tray, which has a larger overall impact. Students are frequently aware of the first effect, since they associate large Reynolds numbers to large Sherwood values, but neglect the second and more dominant effect in this case.

The experimental and predicted overall efficiencies are listed in Table 1, and show that both correlations underestimate  $E_{ov}$ . Students frequently get disappointed with such diverging results. Instructors notice that students almost always doubt their own experimental results, tending to accept without hesitation model predictions. At this point it is essential to keep in mind that the overall column efficiency is a complex function of system properties, operating conditions, and column geometric variables, and that common empirical correlations take only some system properties into account, as is the case of Eqs. (5) and (6) adopted here. Students should be encouraged to search data for similar systems to see that data are frequently 10 to 20% higher than O'Connell's predictions.<sup>[11]</sup>

The results obtained for the continuous rectification at partial reflux ( $P = 125$  W and  $R = 6$ ) are given in Table 2 and Figure 2b. As may be observed, the overall efficiency achieved is about the same of that obtained at total reflux for the same power (92.0% vs. 92.1%). Furthermore, the separation achieved now ( $0.234 \rightarrow 0.685$ ) is inferior to that obtained at  $R = \infty$  ( $0.273 \rightarrow 0.864$ ; see Table 1), which is the expected result for all students.

Figure 3 shows the evolution of both distillate and residue molar compositions during the batch rectification ( $P = 125$  W and  $R = 6$ ). As expected, the cyclohexane content of the residue approaches zero since it is the lighter component.

The fraction of undistilled liquid in the flask,  $B/F$ , was determined by numerical integration of the Rayleigh equation,

<b>TABLE 2</b> Results for Continuous Rectification Experiment at $P = 125$ W and $R = 6$				
$T_D$ (°C)	$T_B$ (°C)	$x_D$	$x_B$	$E_{ov}$ (%)
87.8	93.5	0.685	0.234	92.0

<b>TABLE 3</b> B/F Fraction Obtained by Rayleigh Equation and Mass Balance Results for Experiment at $P = 125$ W and $R = 6$		
$x_D$ (RI)	B/F (Rayleigh Eq.)	B/F (mass balance)
0.410	0.696	0.667

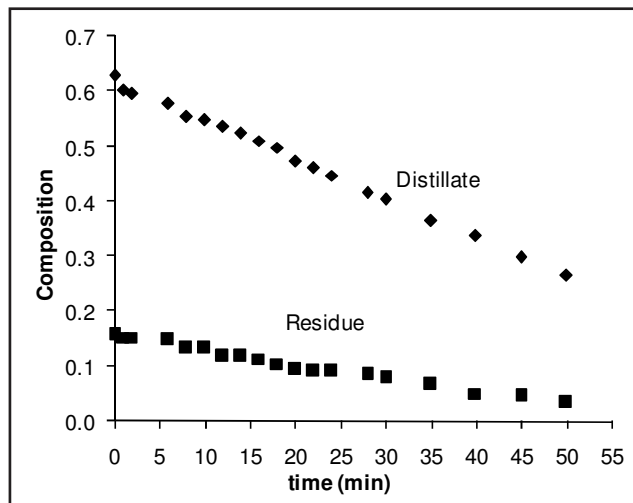


Figure 3. Distillate and residue compositions during the batch rectification at  $P = 125$  W and  $R = 6$ .

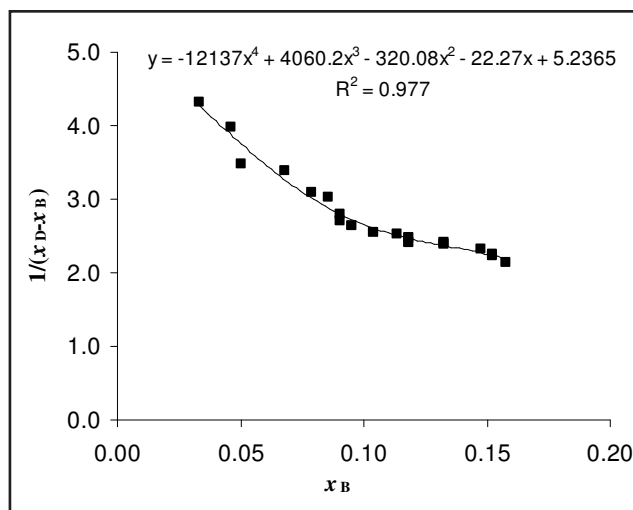


Figure 4. Numerical data used for the integration of Rayleigh equation.

using a polynomial fitted to experimental data (see Figure 4). Many times students are not aware of the impact that the fitted equation has upon the numerical solution. For instance, some groups try to integrate by the trapezoid rule, which gives rise to scattered positive and negative data.

Students calculate  $B/F$  also by mass balance using the initial ( $x_{B,0}$ ) and final ( $x_{B,final}$ ) residue compositions, and the average composition of distillate determined by refractive index. The results found are frequently very similar. In this run (see Table 3) they found  $B/F = 0.696$  and  $0.667$  using the Rayleigh and mass balance approaches, respectively.

## ASPEN SIMULATIONS

Distillations at total reflux and partial reflux ( $P = 125$  W) may be simulated using BatchSep 2006.5 by AspenTech, Inc., a simulator frequently used in industry.



This software allows the simulation of distillation columns under different operating conditions and modes of operation. The embedded VLE calculations were based on the RK-SOAVE method.

### Total Reflux Simulation

The total reflux simulation is carried out using the input specifications and additional information shown in Table 4. For this case, the column is assumed to be initially filled with nitrogen, therefore a partial condenser has to be selected in order to purge it from the system. A feed stream was imposed dur-

TABLE 4 Information for Aspen Simulation at Total Reflux	
<b>Input Specifications</b>	
-	Column initially empty ( <i>initially filled with N<sub>2</sub></i> )
-	Partial condenser
-	Feed stream to introduce the initial charge of mixture
-	Null distillate flow to get $\infty = R$
<b>Additional Information</b>	
-	Column configuration ( <i>number of stages, including reboiler and condenser</i> )
-	Reboiler geometry ( <i>dimensions and jacket type</i> )
-	Power (P = 125 W)
-	Condenser specifications ( <i>pressure, type, area, condensing coefficient, coolant inlet temperature, coolant mass flow, and coolant heat capacity</i> )
-	Tray specifications and dimensions
<b>Operation Steps</b>	
-	i) Column charge
-	ii) Distillation at $\infty = R$
<b>Results</b>	
-	Column holdups
-	Pressure drop
-	Composition profile
-	Temperature profile

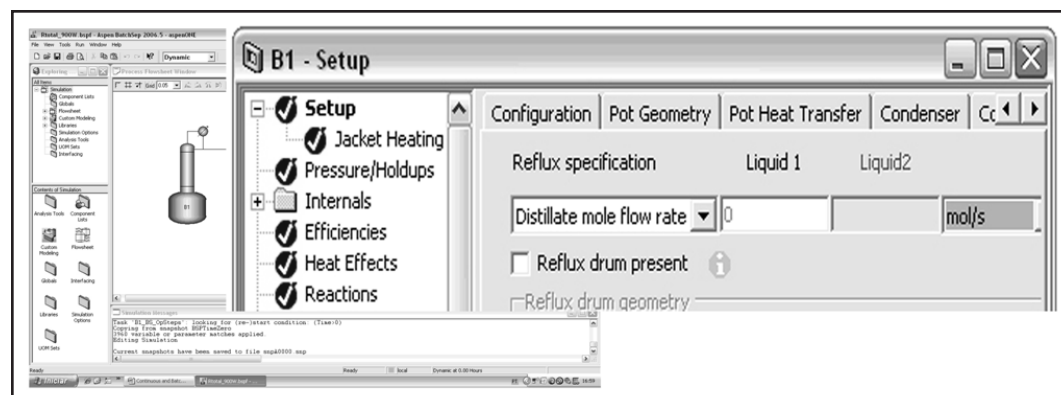


Figure 5. Detail of an Aspen BatchSep 2006.5 window for the total reflux simulation.

ing a predetermined time to charge the tower with the same number of moles that our Oldershaw column contains initially. Subsequently, a null distillate flow must be imposed to reach total reflux condition (see Figure 5). The simulation is carried out in two consecutive steps: i) column charge and ii) distillation at total reflux. Table 5 compiles the pertinent data and options selection for the total reflux calculations, in order to help students to reproduce our results.

### Simulation of Continuous Rectification at Partial Reflux

The continuous rectification at partial reflux ( $R = 6$ ) is computed with the input specifications and additional information compiled in Table 6 (page 112). For this simulation, the column has to be initially at total reflux and only then submitted to  $R = 6$ . Students should realize this approach is in accordance with industrial columns start-up: distillation towers are frequently started up at total reflux, after an initial charge of feed, and this condition runs until both distillate and bottom compositions reach the desired project specifications; only then is the finite reflux ratio implemented.<sup>14</sup> In our case,  $R = 6$ , the column holdups and pressure drop values are those obtained previously from the total reflux simulation, and the feed stream is the distillate recycled to column (see Figure 6, page 112). Table 7 (page 113) compiles data and options for the continuous rectification at partial reflux calculations.

### Simulation Results

The simulation results, presented in Table 8 (page 113) for both total and partial reflux, are in good agreement with the measured values; the relative deviations found lie between 1.0 and 19.3%, being higher for  $R = 6$ . The calculated separation for  $R = \infty$  ( $x_D - x_B = 0.584$ ) is very near the experimental one ( $x_D - x_B = 0.591$ ) whereas it diverges for  $R = 6$  (0.484 against 0.451, respectively). It is curious to notice that students usually doubt their experimental observations against the simulated results, suggesting possible experimental errors for the deviations found for  $R = 6$ . Nonetheless, in this case such large error may be attributed to the fact that some operating parameters, including pressure drop and holdups, were calculated at  $R = \infty$  and assumed to be the same in the continuous partial reflux simulation. On the whole, students and instructors are amazed with simulation results due to the large number of input parameters and specifications, particularly those for geometrical variables.

## CONCLUSIONS

This work describes an experiment in which students have the opportunity to study distillation, using an Oldershaw tray column, under three different modes of operation: total reflux, continuous partial reflux, and batch with constant reflux. The effect of the internal molar flows on column

**TABLE 5****Specification and Options Selection for the Total Reflux Simulation Carried Out With Aspen Batchsep 2006.5**

Window	Tab	Specifications/Selections
<b>Setup</b>	Configuration	Number of stages: 7 Valid phases: Vapor-Liquid
	Pot Geometry	Pot orientation: vertical Pot head type: Top Hemispherical, bottom Hemispherical Diameter: 0.18m Height: 0.18m
	Pot Heat Transfer	Jacket: Heating, Jacket covers head Top height: 0.08m
	Condenser	Condenser type: Partial Partial condenser spec: Coolant temperature Condensing coefficient: 100 cal/hr/m <sup>2</sup> Area: 0.15 m <sup>2</sup> Coolant inlet temperature: 18 °C Coolant mass flow: 100 kg/hr Coolant heat capacity: 4.18 kJ/kg/K
	Reflux	Distillate mass flow rate: 0 kg/hr
<b>Jacket Heating</b>	Jacket Heating	Heating option: Specified duty Duty: 0.125 kW
<b>Pressure/Holdups</b>	Pressure	Pressure profile and holdups: Calculated
<b>Internal 1</b>	Specification	Section: Start stage: 2 End stage: 6  Tray Specifications: Diameter: 0.03m Spacing: 0.025m Weir height: 0.005m Lw/D: 0.83 % Active area: 90 % Hole area: 15 Discharge coefficient: 0.8
<b>Initial Conditions</b>	Main	Initial condition: Empty Initial temperature: 20 °C Initial pressure: 1.01325 bar
<b>Charge Stream Feed</b>	Main	Charge stage: 7 Valid phases: Liquid-Only Feed convention: On-stage Type: Fresh feed Flow rate basis: Mole  Conditions: Temperature: 20 °C Pressure: 1.01325 bar  Composition: Composition basis: Mole-Frac CYCLO-01: 0.3 N-HEP-01: 0.7 N2: 0
<b>Operating Step Charge</b>	Changed Parameters	Location: Charge stream/Feed Charge stream/Feed/Mole flow rate: 0.75 mol/min Jacket/Heating/Duty: 0 kW Condenser/Coolant mass flow: 0 kg/hr
	End Conditions	Step end condition: Elapsed time Duration: 10 min
<b>Operating Step Distill</b>	Changed Parameters	Location: Charge stream/Feed Charge stream/Feed/Mole flow rate: 0 mol/min Jacket/Heating/Duty: 0.125 kW Condenser/Coolant mass flow: 100 kg/hr

performance was investigated at total reflux by changing reboiler power.

Results show that the efficiency decreases slightly with increasing flows. Moreover, column efficiency measured at partial reflux is analogous to that obtained at total reflux. For batch distillation, the application of the generalized Rayleigh equation provides good results. The results at infinite reflux and for the continuous rectification at partial reflux were compared with those obtained by Aspen BatchSep simulations, giving rise to relative deviations between 1.0 and 19.3%.

With this work students practice relevant concepts, including vapor-liquid equilibrium, continuous vs. batch operation, McCabe-Thiele graphical method, and column efficiency as

well as data analysis with the generalized Rayleigh equation. Furthermore, they are introduced to the use of simulation software, an important tool for their chemical engineering instruction.

## ACKNOWLEDGMENTS

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## NOMENCLATURE

B	Final number of moles of liquid in the reboiler, mol
$E_{ov}$	Overall efficiency, %
F	Initial number of moles of liquid in the reboiler, mol
$N_{real}$	Number of real trays
$N_{ideal}$	Ideal number of equilibrium stages
P	Reboiler power, W
$P_t$	Total pressure, atm
$P^o$	Vapor pressure, atm
R	Reflux ratio
RI	Refractive index
T	Temperature, °C
x	Molar fraction of liquid phase
y	Molar fraction of vapor phase

### Greek letters

$\alpha_{12}$	Relative volatility
$\gamma$	Activity coefficient
$\mu$	Molar average liquid viscosity, cP

### Subscripts

B	Bottom
D	Top
final	Final condition
i	Component i
0	Initial condition

TABLE 6 Information for Aspen Simulation of the Continuous Rectification at Partial Reflux	
<b>Input Specifications</b>	
-	Column initially at total reflux
-	Total condenser
-	Total initial charge and composition
-	Distillate flow to get R = 6
<b>Additional Information</b>	
-	Column configuration (number of stages, including reboiler and condenser)
-	Reboiler geometry (dimensions and jacket type)
-	Power (P = 125 W)
-	Column pressure drop and tray holdups
-	Distillate charge stream (charge stage, type, temperature, pressure)
<b>Operating Steps</b>	
-	Distillation at R = 6
<b>Results</b>	
-	Composition profile
-	Temperature profile

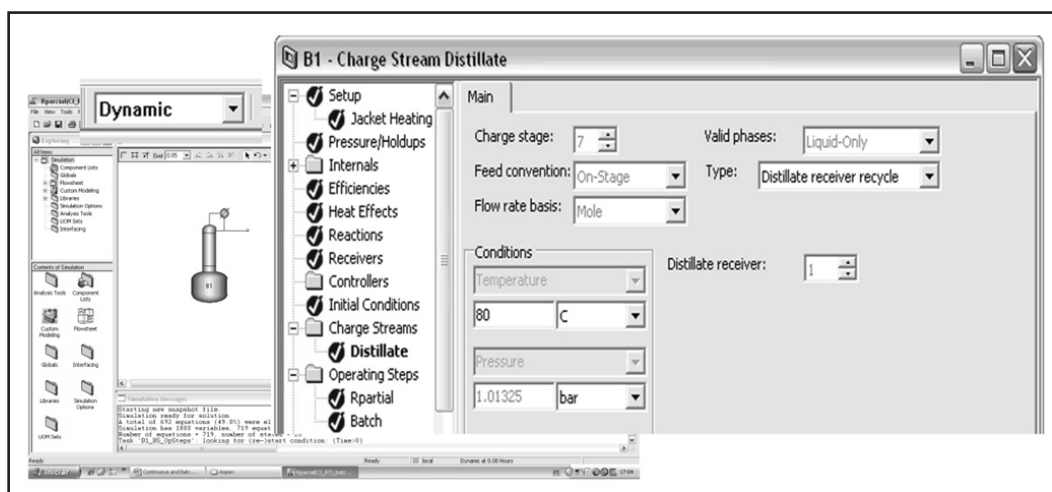


Figure 6. Continuous partial reflux simulation flowsheet.

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**TABLE 7**  
**Specification and Options Selection for the Continuous Rectification at Partial Reflux Simulation Carried Out With Aspen Batchsep 2006.5**

Window	Tab	Specifications/Selections
<b>Setup</b>	Configuration	Number of stages: 7 Valid phases: Vapor-Liquid
	Pot Geometry	Pot orientation: vertical  Pot head type: Top Hemispherical, bottom Hemispherical Diameter: 0.18m Height: 0.18m
	Pot Heat Transfer	Jacket: Heating, Jacket covers head Top height: 0.08m
	Condenser	Condenser type: Total
	Reflux	Reflux ratio: 6
<b>Jacket Heating</b>	Jacket Heating	Heating option: Specified duty Duty: 0.125 kW
<b>Pressure/Holdups</b>	Holdups	Holdup basis: Mole Start Stage: 2 Stage Holdup: 5E-5 kmol
<b>Initial Conditions</b>	Main	Initial condition: Total reflux Initial drum liquid volume fraction: 0.5 Initial temperature: 20 °C Initial pressure: 1.01325 bar
	Initial Charge	Composition basis: Mole-frac Total initial charge: 0.0075 kmol CYCLO-01: 0.3 N-HEP-01: 0.7
<b>Charge Stream Distillate</b>	Main	Charge stage: 7 Valid phases: Liquid-Only Feed convention: On-stage Type: Distillate receiver recycle Flow rate basis: Mole  Conditions: Temperature: 80 °C Pressure: 1.01325 bar  Distillate receiver: 1
<b>Operating Step Rpartial</b>	Changed Parameters	Location: Charge stream/Distillate Charge stream/Distillate/Mole flow rate: 0.1 mol/s Liquid distillate receiver: 1 Condenser pressure: 1.01325 Jacket/Heating/Duty: 0.125 kW

**TABLE 8**  
**Total Reflux and Continuous Rectification Simulations Results**  
Bracketed values are relative deviations to the experimental ones.

	T <sub>D</sub> (°C)	T <sub>B</sub> (°C)	x <sub>D</sub>	x <sub>B</sub>
Total reflux (R = ∞)	83.8	92.1	0.873 (1.0%)	0.289 (5.9%)
Cont. rectification (R = 6)	85.6	92.4	0.774 (13.0%)	0.290 (19.3%)