Separation Process Engineering

Includes Mass Transfer Analysis

Fifth Edition

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IV. Plotting McCabe-Thiele Diagram from Aspen Plus Simulation for Assignment

13. While in the lab, print out the y-x graphs that you will need to solve Problem 8.G6 (MEK versus water [plot MEK mole fractions] at 1.0 atm and water versus MEK [plot water mole fractions] at 100.0 psia). In addition, print the tables that give liquid and vapor flow rates for both columns. Read Problem 8.G6 before you close Aspen Plus to be sure you have all values needed.
14. **Assignment:** Solve Problem 8.G6.

    Answers: Step 4: Because F and B_1 are specified, specifying B_2 will over specify the system.

    Step 7: Because $B_2 = F - B_1$.

Lab 8. Binary Distillation of Systems with Heterogeneous Azeotropes

**Answers are at the end of Lab 8.*

Preparation

- Review computer Labs 1 to 6 as needed.
- Review convergence procedures for systems with loops (Lab 7).
- Carefully review convergence procedures in Figure 6-1.
- Read Sections 8.2.1 and 8.2.3.

Goal: Use Aspen Plus simulation to explore separation of binary systems that have a heterogeneous azeotrope.

The purpose of this lab is to design systems similar to Figures 8-3A and 8-5A for separating n-butanol and water.

Preliminary

1. Open a new, blank file. Under Setup, set valid phases as Vapor-Liquid-Liquid. In the Selection tab of Components, list n-butanol and water as the components. Aspen recognizes the component ID butanol as n-butanol.
2. Finding a suitable VLE model for heterogeneous azeotropes is a challenge. NRTL-RK was the best model of the half dozen I tried. NRTL-RK fits the vapor composition of the azeotrope and the liquid composition of the water phase quite well (see Table 8-5) but misses a bit on the butanol phase composition. (Feel free to fit the NRTL-RK constants using the method in Appendix B at the back of the book.)
3. Use Analysis to look at the T-y,x and y-x plots. If you get a pop-up message “No such file or directory” after you click the Run Analysis button, click OK, wait a few seconds, and the T-y,x plot will appear. Compare with the data. Remember to check that Vapor-Liquid-Liquid are the valid phases in Analysis.

Part I. Single-Column System

4. In simulation mode, draw a diagram similar to Figure 8-3A except include a total condenser with a reflux stream as part of the RadFrac column. The distillate product is a liquid, and this line is connected to the decanter (or liquid-liquid settler) feed (listed in Model Palette under Separators). The hydrocarbon layer from the decanter (first phase) should be connected to the distillation

column feed. The aqueous layer (second phase) from the decanter is the water product. The flowsheet for the one-column system is shown in Figure 8-A2. Note that the Aspen Plus column in Figure 8-A2 does not look exactly like the arrangement in Figure 8-3A. We are initially using the condenser on the column to condense the liquids. We will use specification sheets to make the system behave like Figure 8-3A.

5. The feed is 100.0 kmol/h of a saturated liquid feed at 1.0 atm pressure. The feed is 78.0 mol% n-butanol and 22.0 mol% water.
6. Column: Calculation Type: Select equilibrium from menu.
   - **a.** Start with the number of stages N = 10.
   - **b.** Use a total condenser and a kettle-type reboiler.
   - **c.** Select Vapor-Liquid for valid phases because best practice is to avoid having two liquids in the distillation column—handling two liquids is the job of the decanter.

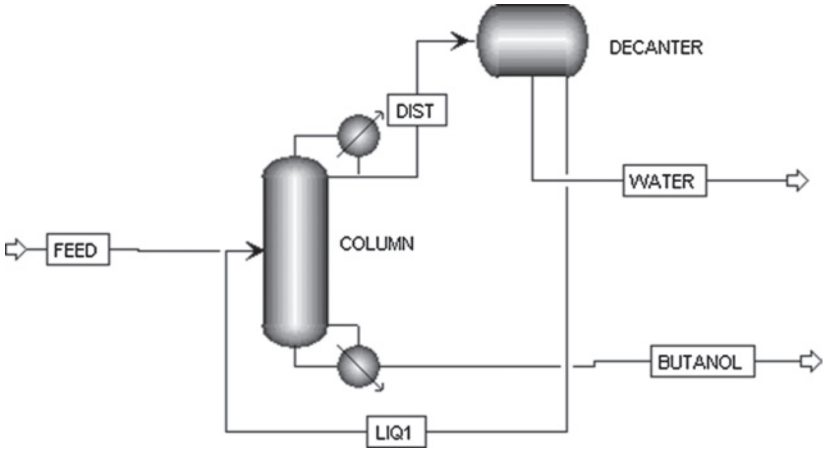

FIGURE 8-A2. *Single-column distillation system for separation of heterogeneous azeotrope*

   - **d.** Select convergence as the strongly nonideal liquid.
   - **e.** Set the reflux ratio to 0.5. We do not really want to reflux from the condenser, but we do want to take our reflux from the decanter; however, if you try setting the reflux ratio to 0.0, Aspen Plus will not run. Once Aspen runs, in step 8 reduce the reflux ratio in a few steps down to about 0.025. This ratio is small enough that results will be very close to using only reflux from the decanter phases. If you do not do this in steps, the condenser will dry up, and the run will have errors. Pamper Aspen Plus, and make it happy! After you finish the lab, you might try reinitializing, and see what happens if you set the reflux ratio to 0.025 immediately. Results when the column or condenser is dried up are not useable.
   - **f.** Specify a *bottoms flow rate* that will give you the desired purity of the butanol product (0.990 or higher mole fraction). Purity of the water product from the decanter depends on the VLE correlation used. Initially, use the value from Table 8-5 and then correct it once you have the decanter results. The mass balance to calculate the bottoms flow rate has to be accurate.
   - **g.** Initially, specify the feed location NF = 5 as a liquid.
   - **h.** Specify that the line from the decanter is input in the distillation column at $N_{decanter} = 2$ as a liquid.
   - **i.** Operate at a pressure of 1.0 atm.
   - **j.** If you have convergence difficulties in step 8, first verify that you checked Strongly non-ideal liquid for the column. If you still have difficulties, in the convergence section of the column block (in the navigation pane), click Convergence and in the window click the Basic tab. Then set the Maximum iterations to 75. Higher values usually do not help.

7. Decanter. Pressure is 1.0 atm, and the heat duty is 0.0 (adiabatic). The key component to identify the second phase is water (click water and then click the > button), and set the threshold at ~0.7 (equilibrium data show the highest water content in the organic phase is <0.6). If you have difficulty with decanter convergence, click Calculations Options and Convergence parameters, and set Maximum iterations at 75. If that does not help, reduce the decanter's Error tolerance (delete one zero).
8. Run the simulation. If the water product purity* is different than the value used for mass balance to determine the bottoms flow rate, redo the mass balance. Run the simulation again with the revised B value in the Column Configuration window. Then return to step 6e and reduce the reflux ratio in steps.
9. Record mole fractions, temperatures, and flow rates (kmol/h) of the butanol* and water product streams, the hydrocarbon layer refluxed to the column from the decanter (LIQ1 in Figure 8-A2), and distillate.*

Part II. Two-Column System

For Part II the purity of both products is 99.0 mol% or higher.

10. Recalculate the bottoms flow rate for the distillation column based on the new water product purity. Adjust the first column's condition.
11. Modify your flowsheet to include a stripping column (Figure 8-5A), except use the column condenser to produce a liquid distillate. Your flowsheet should be similar to Figure 8-A3.

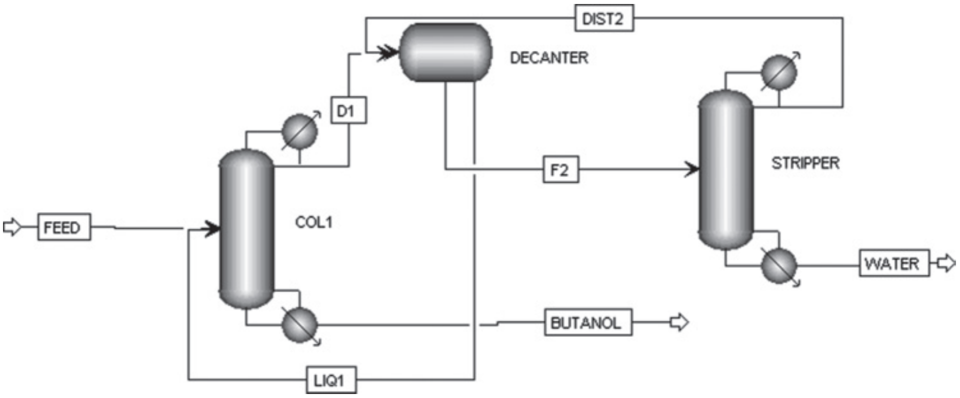

FIGURE 8-A3. *Two-column distillation system for separation of binary heterogeneous azeotrope*

12. Stripping column specifications: Calculation Type: Select equilibrium from the menu.
    a. Initially set the number of stages to N = 10.
    b. Use a total condenser and a kettle-type reboiler.
    c. Select Vapor-Liquid for the valid phases.
    d. Select convergence as Strongly non-ideal liquid.
    e. Set the reflux ratio to 0.5. Reduce the reflux ratio in a few steps down to about 0.025.
    f. Set the boilup rate to 1.0 kmol/h in the stripping column. This is *rate,* not boilup *ratio*.
    g. The line from the decanter should be input as feed on stage 2 (first actual stage in the column). If you have convergence difficulties, see instruction step 7.
    h. Pressure is 1.0 atm.

13. Run the simulation for a two-column system. If one or both columns are not producing products as pure as required, increase the boilup rate in the stripper modestly in increments of 0.2 kmol/h until both products meet the purity specifications.
14. If the butanol product is purer than necessary, this implies column 1 can have fewer stages and/or a lower boilup ratio. However, before changing the conditions, save your results, and note that there is an inherent difficulty in the set of specified variables for the flowsheet in Figures 8-A2 and 8-A3. The loop from Col-1 to the Decanter and back to Col-1 via the HC-Layer does not have control on flow rates. (The other loop from the Stripper to the Decanter and back does because the boilup rate in column 2 is specified.) Thus, it is possible for the flow rates of Distil-1 and HC-Layer to become very large. This will happen if the number of stages in Col-1 are reduced drastically. The cure is to set a flow rate that will control this loop; however, since the number of variables you can specify is set, another variable has to be made free. A method that works once Aspen Plus is giving good results is to set the reflux ratio (0.025) and the boilup rate in Col-1 (use the value from the run with 10 stages and $N_{feed} = 5$ with bottoms in Col-1 specified). To be sure this works, repeat the previous run but with the boilup rate in Col-1 replacing the specification of the bottoms rate. Then reduce the number of stages and the feed location. Increase Col-1 boilup rate by a maximum of 10.0% to obtain the desired separation. If there are convergence difficulties as you do this, try decreasing the error tolerance for each column to 1.0 E-5 (for each distillation block, scroll down to and click Convergence, then click the subheading Convergence. In the window, click the Basic tab and change the Error tolerance to 0.00001. Also try increasing the number of maximum flowsheet iterations in the Wegstein method to 75—scroll below Block, Utilities, and Reactions in the Navigation pane [All Items] and click Convergence → Convergence Options → Methods → Wegstein tab → Convergence Parameters → Maximum flowsheet evaluations).
15. Return to the stripper and see if you can reduce the number of stages* and perhaps adjust the boilup rate* to slightly lower values. Although both product purities are affected by changes in the stripper, the butanol is affected more. Be sure to keep product purities* ≥0.990.
16. Report your results for the two-column system: *list the number of stages in both columns, optimum feed locations, boilup rates in both columns, and product purities.*
17. When done, make the two-column system not converge! Return to the original number of stages, the original feed location, and the original specification of the bottoms flow rate in the butanol column. Increase the boilup rate in the water stripper to 10.0 kmol/h, and run the simulation. Messages in the control panel and the run summary and the reports for the blocks all state there is a problem. However, the stream reports do not state there is a problem. Do NOT save runs that do not converge.

Answers: Part I. Step 8. Butanol mole fraction water layer from decanter = 0.0249 and with reflux ratio decreased to 0.025 butanol mole fraction was 0.0247.

Part I. Step 9. Butanol product purity 0.99006, flow rate 78.24 kmol/h, T = 116.4°C. Distillate: 45.342 kmol/h, 0.29598 mole fraction butanol.

Part II, Step 15. Stripper Number of stages = 4. Stripper boilup rate = 1.55 kmol/h. Stripper purity = 0.99004.

Lab 9. Simulation of Extractive Distillation

Preparation:

- Review computer Labs 1 to 6 as needed.
- Review the convergence procedures for systems with loops (Labs 7 and/or 8).
- Carefully review the convergence procedures in Figure 6-1.
- Read Section 8.6.

Goal: Use Aspen Plus simulation to explore the application of extractive distillation as a method to separate azeotropic mixtures.

This assignment is more prescriptive and involves less exploration than other labs because convergence can be difficult with extractive distillation. The basic algorithm (Figure 6-1) assumes the concentration loop will have little effect on the other loops. Extractive distillation systems have very nonideal VLE, concentrations have a major effect on K values, and a concentration loop is added to Figure 6-1. As always with Aspen Plus, it may be possible to obtain convergence by starting with a set of conditions that converges and slowly changing the variable of interest (e.g., D) to approach the desired value. When you have a convergence problem, first reread the instructions to be sure you have properly set convergence parameters. Next, try increasing the number of iterations in the block (your distillation column) convergence by 1 (this is done so that Aspen Plus will run again) and then run it again. Second, a modest reduction in error tolerance in the convergence may help—then run it again. Third, return to a condition that converged previously and run it. If this condition does not run, reinitialize Aspen Plus and run the known conditions that converged. Once Aspen Plus is running, work your way toward the desired condition by changing the desired variable (e.g., D) by modest amounts.

Use Strongly non-ideal for convergence of both columns.

Standard convergence will not work.

I. Desired specifications

In Figure 8-14, the A product will be the ethanol product, and the B product (distillate from column 2) will be water.

- **A.** The ethanol product must be at least 99.75 mol% ethanol (this exceeds the requirements for ethanol used in gasoline).
- **B.** The bottoms product from column 1 should have less than 0.009 mol% ethanol. This number is low to increase the recovery of ethanol.
- **C.** The distillate from column 2 should contain less than 0.01 mol% ethylene glycol (EG). This value is low to reduce the biological oxygen demand (BOD) of the water.
- **D.** The bottoms product from column 2 should contain less than 0.01 mol% water because water in this stream will probably exit in the ethanol product when the solvent is recycled.

II. Set up system and flowchart

1. In Setup, the valid phases are Vapor-Liquid. To use extractive distillation to break the ethanol-water azeotrope, input the components ethanol, water, and ethylene-glycol (Aspen also recognizes EG). For VLE use NRTL. After running property/analysis setup, click Residue Curves (right side of the Home toolbar) for the system at a pressure of 1.0 atm. Make the water component 1, the ethylene glycol component 2, and the ethanol component 3 (you will need to override Aspen Plus to do this). Run the analysis with 10 to 15 curves. Compare your result with Figure 8-15.
2. In Simulation mode, draw the two-column system as shown in Figure 8-A4, but do not connect the RCYCLSOL stream to the COOLER (leave it as a product initially). Both columns should be drawn with liquid distillate products. The flowsheet will be similar to Figure 8-A4, except the RCYCLSOL STREAM is temporarily a product.

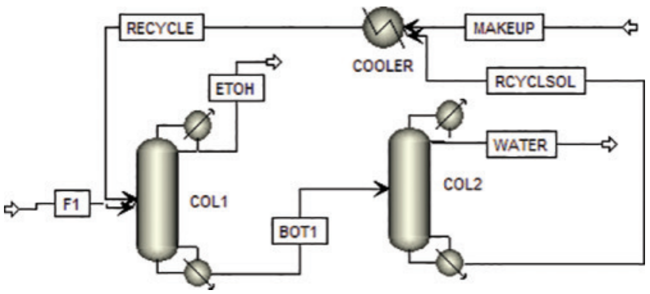

FIGURE 8-A4. *Two-column distillation system for extractive distillation*

3. The feed to column 1 is 100.0 kmol/h of a saturated liquid at 1.0 atm pressure that is 74.0 mol% ethanol and 26.0 mol% water.
4. Initially, the makeup solvent is a second feed to column 1. Makeup will eventually be pure ethylene glycol at 1.0 atm pressure. However, initially use a makeup solvent that is close to the expected concentration of the recycle solvent. That is, the makeup solvent stream should be 99.99 mol% ethylene glycol and 0.01 mol% water. Use a solvent temperature of 80.0°C to approximately match the temperature of the solvent feed stage (NS = 5) in column 1. Use an initial makeup solvent rate of 52.0 kmol/h.
5. *Heat exchanger*. Eventually, we will connect the solvent recycle loop from column 2 to column 1. First, include a heat exchanger to cool the solvent. If you do not cool the solvent, column 1 will not work after you connect the solvent recycle loop. (Why not?) (To put a heat exchanger in the MAKEUP line, go to Heat Exchangers and put a Heater—used and labeled as a COOLER in this case—in your flowsheet. Then left-click the MAKEUP stream, and right-click Reconnect Destination. Connect the MAKEUP stream to the heat exchanger inlet arrow. Click Next. Connect the RECYCLE line to the outlet of the cooler and to the feed port of Col1. In the heat exchanger window, use 80.0°C, pressure = 1.0 atm; valid phases are liquid only.) We will hook up the RCYCLSOL stream from column 2 in step 12.
6. *Column 1.* Calculation Type: Select equilibrium from the menu.

   a. Start with the number of stages at $N_1 = 50$.
   b. Use a total condenser and a kettle reboiler
   c. Select Vapor-Liquid for valid phases.
   d. Select the convergence as Strongly non-ideal liquid.
   e. Use the external reflux ratio in column 1 of L/D = 1.0. (Normally, L/D would be optimized, but to save time, leave it constant.) The reflux is returned as a saturated liquid.
   f. Specify the distillate flow rate that will give you the desired purity of ethanol product and the desired ethanol mole fraction in bottoms product (do external balances). The mass balance to calculate distillate flow rate has to be accurate.
   g. Initially, specify the feed location as NF1 = 23.
   h. The solvent is treated as a second feed to column 1. In the flow diagram, use the feed port to add a solvent feed (RECYCLE) to the column. Then specify its location (NS = 5) in the Specifications for the column Block in the Streams tab.
   i. Operate at a pressure of 1.0 atm.
   j. Scroll down Col1 to Convergence. Click Convergence and then the second Convergence. Click the Basic tab, and in the menu for Maximum iterations, increase the number to 75. This set of conditions should remove sufficient ethylene glycol from distillate to produce an ethanol of suitable purity. We will check later to make sure that this happens.

7. *Column 2.* In the flow diagram, the feed to column 2 is the bottoms from column 1.

   a. Calculation Type: Select equilibrium from menu.
   b. Start with the number of stages as N2 = 50 to find $(L/D)_{min}$ for column 2 in step 10.