Robust and Reliable Design Method of a Distillation Column

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Abstract

Many industrial processes and plants today use distillation as a form of solvent recovery by separating mixed solvent streams into their respective components. Distillation design can be a complicated and costly process. Therefore, when designing a column separation process it is imperative to know whether your design is going to work up to your specifications. You want to be able to determine this as quickly and efficiently as possible to save both time and money. Thus this problem is addressed at the laboratory for product and process design.

Design of a distillation separation can be a both time consuming and difficult problem. One cannot predict apriori whether the design for a column is feasible given a set of specifications. Simulators, such as HYSYS and ASPEN predict feasible designs by trial and error, but do not converge if the specifications are infeasible. Here in the laboratory of product and process design we have developed a feasibility test for a distillation separation given a certain set of specifications. This new program, written by Libin Zhang, will provide information on whether the design is feasible or infeasible in a matter of seconds. This design is then simulated in HYSYS to ensure the accuracy of the results outputted from the new feasibility test software. Tests are run on both ideal and non-ideal ternary and quaternary mixtures. Following those trials, a column sequencing test was then run. The feasibility design testing program uses a continuous temperature collocation calculation method to solve for the profiles.
Introduction

Distillation is a method of separating a mixtures of components based on their volatilities. One advantage of distillation is that it is a cheap and simple method of separating components. It can also separate components are a much larger scale then other methods. Distillation is used to recover solvents from a reactor, separate these solvents and then they may be recycled back into the process or sold off at a lower purity. This allows a plant to save money on solvents and provide other industries with the components they need in their processes. Distillation is used in the beer, wine, pharmaceutical, petroleum, coating, flavoring, and wastewater industries as well as many others. As stated it is an integral part of production of everyday products.

Distillation falls into two main categories: continuous and batch distillation. Continuous distillation is a process that takes a mixed feed stream and separates its components into two or more productions. In batch distillation takes in much feed at once and then removes the more volatile components over time. The main focus of this paper will be on continuous distillation. These two distillation methods can be subcategorized into ways these methods can be achieved. These ways include vacuum distillation, in which the pressure is lowered to separate components with very high volatilities; reactive distillation, in which a reaction takes place to separate the two components; and azeotropic distillation, in which an entrainer must be added to help get a more pure separation between components.

Part 1 of this report will discuss continuous distillation columns and their design. Part 2 will present the design and simulation results from the trials. Part 3 will explain
the results for the column sequencing model and the final part will draw conclusions and
discuss future work.

1 Continuous Distillation Column Design

A basic overview of a continuous distillation consists of cylindrical column with
stripping and rectifying sections, a condenser, and a reboiler. The streams include one or
more feed streams and distillate and bottoms streams. The stripping section of the
column removes the light materials from the heavy ones and in the rectifying section the
heavy material is removed from the light components.(Figure 1a) The column is filled
with a certain number of trays or stages at which the separations are taking place.
Theoretically, the vapor and liquid at each stage are in equilibrium.(Figure 1b)

![Diagram of continuous distillation column](image1.png)

**Figure 1-a Diagram of continuous distillation column**

![Close up of stage of continuous distillation column](image2.png)

**Figure 1-b Close up of stage of continuous distillation column**

1.1 McCabe-Thiele Diagrams
For a distillation column to work up to the desired performance it must be designed to achieve certain specifications. This design can be done graphically. Both the stripping and rectifying profiles can be represented geometrically in terms of their component compositions. One method of representation is the McCabe-Thiele diagram. Using a vapor equilibrium curve diagram, one can graph the stripping and rectifying profiles linearly as functions of the reflux and reboil ratios. At the intersection of these profiles is the feed point. The number of stages required can then be drawn in. (Figure 2) Unfortunately this design method can only be used for binary systems and systems with constant volatilities. Three component systems, ternary systems, require profiles that are non-linear. Thus another method must be used to design separations of three or more component systems, both ideal and non-ideal. Underwood’s equations are difficult to solve and fail to converge when the specifications are infeasible. So we must find a more reliable and simple method of finding the profiles.

1.2 Boundary Value Method

The method that is used by the feasibility design test program written in Delphi is similar to the boundary value method (BVM), introduced by Levy and Doherty in 1985, in that it uses the intersections of the profiles to predict feasibility. It can be used for both
ternary and quaternary separations. It uses a continuous calculation method to find the column profiles as opposed to the tray by tray calculation used by the HYSYS simulator. Feasibility design of a column can then be determined graphically by the intersection of the stripping and rectifying profiles in composition state space. The point of intersection tells the user where the liquid feed tray occurs. (Figure 3a)

To determine the minimum reflux ratio for a design, one chooses a reflux ratio in which the intersection of the two profiles is possible. One profile should terminate when it touches the other profile. (Figure 3b)

As mentioned above the calculation method uses a tray by tray method of computing the profiles. The software developed by Dr. Libin Zhang uses temperature as a variable rather than height and the number of trays. It is based on the continuous differential equation methods developed by Doherty. These profiles are then determined by using a finite element collocation method. This method will be expounded upon in later paragraphs. The following is Dr. Zhang’s method for designing the algorithm behind the software to determine the feasibility design of a separation.
1.3 Composition Profiles

The first step in determining profiles is doing mass balances around the various stages of the rectifying and stripping sections of the column. When doing this one is to assume constant molar overflow.

1.3.1 Finite Reflux

For any distillation column the overall mass balance is

\[ F = D + B \]  \hspace{1cm} (1)

For any specific component \( i \), the mass balance would be

\[ Fx_i = Dx_i + Bx_i \]  \hspace{1cm} (2)

Since the vapor and liquid above and below each stage is in equilibrium the balance equation to describe a stage in the rectifying section is

\[ x_{i,n-1} = \frac{r+1}{r} y_{i,n} + \frac{1}{r} x_{i,D} \]  \hspace{1cm} (3)

and for the stripping section

\[ x_{i,n+1} = \frac{s}{s+1} y_{i,n} + \frac{1}{s+1} x_{i,B} \]  \hspace{1cm} (4)

where \( i = 1, \ldots, C-1 \).

These equations can be used to develop profiles using a stage by stage computation method. To convert these balances into continuous profile equations we must first expand them to the first term of the Taylor series:

\[ y_{i,n} = y_{i,n-1} + \frac{dx_i}{dh} \bigg|_{h=n} (\Delta h) + \frac{1}{2} \frac{d^2x_i}{dh^2} \bigg|_{h=n} (\Delta h)^2 + ... \]  \hspace{1cm} (5)

where \( \Delta h = (n+1)-n = 1 \)

We may disregard all terms of the Taylor expansion greater than the first derivative.

After obtaining this expansion we may solve for the continuous profile as below for the stripping section:
\[ \frac{dx_i}{dh} = \frac{s}{s+1}y_i - x_i + \frac{1}{s+1}x_{i,B} \]  \hspace{1cm} (6)

And for the rectifying section:
\[ \frac{dx_i}{dh} = x_i - \frac{r+1}{r}y_i + \frac{1}{r}x_{i,D} \]  \hspace{1cm} (7)

1.3.2 Infinite Reflux

For columns that have refluxes and reboil ratios moving towards infinity, equations 6 and 7 must be modified to satisfy this condition. As the reboil ratio goes to infinity the stripping profile becomes
\[ \frac{dx_i}{dh} = y_i - x_i \]  \hspace{1cm} (8)

As one fraction approaches one and the other zero. Similarly rectifying profile transforms into
\[ \frac{dx_i}{dh} = x_i - y_i \]  \hspace{1cm} (9) as the reflux ratio approaches infinity.

1.4 Finite Element Collocation

As mentioned in the latter section of the report, collocation is the method used by the feasibility design testing software. Due to my lack of technical mathematical insight of the specifics of the collocation method, I will try to explain it as best possible as I understand it.

Background

The collocation method is a way of solving differential equations. To begin one must start with a differential equation:
\[
\frac{dy}{dx} = f(x, y), \\
y(0) = y_0.
\]

We then want to solve \( y \) as a polynomial function in \( x \):

\[
y = y_0 + a_1x + a_2x^2 + a_3x^3 + \ldots + a_nx^n
\]

(11)

This can be seen in figure 4.

After obtaining this polynomial form one can solve for the coefficients. After finding the equations the fixed collocation points may be solved for by using residual error. The residual error \( (e_i) \), can be calculated by using the formula

\[
e_i = f(x, y) - (a_1+2a_2x+\ldots na_nx^{n-1})
\]

(12)

For each point the residual error should be very close to or equal to zero. After computing at a large number of collocation points, \( n \), the Lagrange polynomial interpolation method is used to fit a polynomial through the fixed points so that the new polynomial is very close to the true solution.

In the case of the feasibility design software, the differential equations that is used is the composition of a component with respect to temperature. The temperature increases monotonically as one goes up the column. This information is determined by the thermodynamic data. Where the pinch points occur in the column is where the fixed points occur. A continuous composition profile can then be fit to these points resulting in the stripping and rectifying profiles. Similarly, this method can be applied to a distillation column with the variables being the trays and mole fractions at that tray.
2 Design and Simulation

Before continuing, one must first discuss the difference between design and simulation. Design is specifying certain aspects of your column separation such as component recovery, feed, and reflux ratio. When designing a new column separation one does not know apriori whether the design for a column is feasible given a set of specifications. These specifications are the results you would like the column you are designing to have, i.e. a high recovery of one component in the distillate. But when designing a column it may be that it is impossible to obtain a certain separation of components with these specifications are then tested if they are feasible using the feasibility design program.

Simulation is taking computer generated column with a certain number of trays and a given feed and some sort of result can always be obtained, similar to running a pilot plant model of a column. Simulation is much easier to use when the feasibility of a design is already know. Without knowing the feasibility one could try for hours trying to make a column work with no success, if the separation design is impossible to begin with. The purpose of the simulation software is to ensure that a given design can indeed obtain wanted results.

2.1 Feasibility Design Procedure

Before beginning the design and trials and results it is best to explain how the design software are operated. A screen shot of the Feasibility design program written by Libin Zhang is shown below (figure 5).
2.1.1 Behind the Design Tool

The design tool calculates the stripping and rectifying profiles for the column sections using a computer generated algorithm. This algorithm uses the profile equations (6) and (7) that are found by a Taylor expansion series truncated after the first term. These profiles are then converted into continuous profiles using the temperature collocation methods described in section 1.4. The temperature collocation method is used rather than using height as a variable, because when using height an infinite number of stages is required at a pinch point, making the calculation difficult. The temperature can be measured exactly, giving a much more accurate prediction of the pinch points. Applying the temperature collocation method results in the following equations:

Stripping profile:
These equations in turn become graphical representation of the column profiles in the design.

2.1.2 Starting up

The program file is run in Delphi. After opening the program, it will display a screen that looks like the left image of figure 5. The three top most radio buttons are used in selecting the type of mixture one will be designing a column for; in this case it is a non-ideal mixture. The set of radio buttons below the first three are the buttons used in the selections of the mole fractions of the components in the distillate and bottoms as well as the reflux or reboil ratio.

2.1.3 Entering Data

In this case the first radio button is selected. In this instance the user wants to define the mole fraction of .95 of component 1 in the distillate, a fraction of 0.049 of component 2 in the distillate and 0.05 fraction of component 1 in the bottoms, along with

\[
\frac{dx_i^s}{dT} = -\left( \frac{1}{s+1} y_i^s - x_i^s + \frac{1}{s+1} x_i^{s,b} \right) \frac{\sum_{j=1}^c \left( \frac{\partial K_i^s}{\partial T} x_j^s \right)}{\sum_{j=1}^c \left[ \left( \frac{1}{s+1} y_j^s - x_j^s + \frac{1}{s+1} x_j^{s,b} \right) K_j^s \right]}
\]

(13)

Rectifying profile:

\[
\frac{dx_i^r}{dT} = -\left( x_i^r - \frac{r+1}{r} y_i^r + \frac{1}{r} x_i^{r,b} \right) \frac{\sum_{j=1}^c \left( \frac{\partial K_i^r}{\partial T} x_j^r \right)}{\sum_{j=1}^c \left[ \left( x_j^r - \frac{r+1}{r} y_j^r + \frac{1}{r} x_j^{r,b} \right) K_j^r \right]}
\]

(14)
an external reflux ratio of 2.5. He simply clicks on each cell with the mouse and enters
his values mentioned above. The line below the product composition and reflux line, is
the feed composition specifications line. In this case the feed stream consists of 0.3
component 1, 0.3 of component 2, and 0.4 of component 3. It is easiest to use the lightest
key as component 1 the next lightest component 2 and so on. The Antoine coefficients
for each component can then be entered as well as the critical pressure and Wilson
interaction coefficients. The Antoine coefficients and critical pressures are taken from
Henley and Seader’s *Equilibrium-Stage Separation Operations in Chemical Engineering*,
1981. The Wilson coefficients may be obtained from Gmehling, Kolbe, and Onken
*Vapor-liquid Equilibrium Data Collection Vol*. After entering this data one may save his
work by choosing the *save parameter* button as well as load a save design, but using the
*load* button.

**2.1.4 Graphical Simulation**

Now that all required data has been entered, one may choose to simulate the
design but using the *simulate* button. The graphical results in the right of figure 5 may
be obtained by clicking on the *graphical* tab. The rectifying profile will be displayed in
red and the stripping profile in blue. The feasibility is know when these profiles intersect
at a point. These profiles can then be exported to excel to be compared to the results
found using the HYSYS simulator. The design profiles for the minimum and maximum
reflux can also be displayed by pressing the *Rmin* and *Rmax* buttons respectively.

**2.2 Validation of the Design Tool**
After obtaining results from our design program, it is necessary to validate their reliability by using the commercial simulator HYSYS. Since the design software relays feasibility of a design to a user, it is quite easy to enter the same data into the simulator and compare the resulting composition profiles and discover whether this design tool is indeed a reliable method for predicting the design feasibility and recovery composition.

### 2.2.1 Hysys Simulation Procedure

For the trials done in this paper, HYSYS simulator version 2.4 by Hyprotech was used in the simulation of the columns to ratify the design found on the feasibility design software. The HYSYS simulator user interface is user friendly. Upon staring the program a window will appear with a tool bar on it. (figure 6a)

Select File from the menu bar and drag down to select new case. After doing this the simulation basis manager will open and you then select **add fluid package**. The next window will appear (figure 6b). One must then select the properties package you desire. In this case Antoine. After selecting the Package, then click the **components** tab. In the
case of non-ideal mixtures, one would select Wilson package, as is used in the non-ideal trials in this paper, as illustrated in figure 6c. The Wilson coefficients may be entered manually if needed as will be done for the non-ideal trials later. After selecting your components close the fluid package window and then click the **Enter Simulation Environment** button from the simulation basis manager. This will take you to the workbook window. In this window shown in figure 6d, one enters the physical specifications of the feed stream (temp, pressure, composition etc. . .)
After entering this data one can add a unit operation, in this case a distillation column by choosing the Unit Ops button and selecting distillation from the list of units. After selecting the distillation column, the screen will appear as in figure 6e.

![Figure 6e Column specification Window](image)

This window allows the user to enter what the specifications of the distillation column (i.e. streams, trays, etc.. . ). After completing the column specifications, one may start a simulation by pressing run. The column specifications may be monitored and edited by going to the monitor tab (figure 6f). If the simulation is successful there should be a green “converged” next to the reset button. A graphical PFD version of the column flow sheet may be viewed by choosing the PFD button on the tool bar this button is a picture of a small column. A window will appear with the current flowsheet (figure 6g).
2.2.2 Physical Property Entries

To ensure that results are obtained that are accurate between the design and simulation tools, the physical property data that is enter must be identical. In the case of Antoine coefficients for ideal mixtures this was not possible, but the difference is small enough that it may be neglected. Further insight into this topic will be discussed in the Model Vapor Pressures section of the paper. The Wilson activity coefficients were taken from an outside source and manually entered into both tools by the methods prescribed in their procedures.
3 Ideal and Non-ideal Ternary Separation Trials

This paper shall present three ideal and three non-ideal ternary separation trails in the main body.

3.1 Design and Analysis of Ideal Ternary Separations

Ideal 1

The purpose of this trial is to enter the given data from tables 1 and 2 into the design tool as prescribed in the procedure. Upon running the program one receives a graphical representation of the stripping and rectifying profiles. As can be seen in figure 7. These profiles reflect the sections of the distillation column itself and the compositions at each stage. These composition profiles are then compared with the graphical results obtained using the Hysys simulator.

<table>
<thead>
<tr>
<th>Component</th>
<th>Diethyl ether</th>
<th>Hexane</th>
<th>Octane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Composition</td>
<td>0.4</td>
<td>0.25</td>
<td>0.35</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>0.75</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.93</td>
<td>0.067</td>
<td></td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>9.5e-7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2 Antoine Coefficients Ternary Ideal 1

<table>
<thead>
<tr>
<th></th>
<th>Diethyl Ether</th>
<th>Hexane</th>
<th>Octane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diethyl Ether</td>
<td>5.976844</td>
<td>4614.723</td>
<td>388.8028</td>
</tr>
<tr>
<td>Hexane</td>
<td>6.039243</td>
<td>5085.758</td>
<td>382.7941</td>
</tr>
<tr>
<td>Octane</td>
<td>6.4141</td>
<td>5947.491</td>
<td>360.26</td>
</tr>
</tbody>
</table>

Results:

The design results are feasible as observed in figure 7 from the stripping and rectifying curve produced. The stripping and rectifying composition profiles generated by the feasibility design testing program reflect accurately the upper and lower ends of
the column as well as the liquid feed tray composition. One is mainly concerned with the
exiting compositions being very similar and therefore the other parts of the column where
the composition profiles are not so close may be neglected. This difference in their plots
can be attributed to two main sources. One is that HYSYS and the design program use
different Antoine equations to predict the vapor pressure; these differences shall be
discussed in section 3.2 of the paper. Secondly, the design program uses a computes
using a continuous model as mention in earlier sections of this paper, while the HYSYS
simulator uses a tray by tray calculation to find the compositions. Also it should be noted
that the ending points of the profiles and feed point are approximately linear, which
should be the case in any ternary separation.

Figure 7 Results for ideal ternary mixture #1

Ideal 2
Here we will be designing and analyzing an ideal ternary mixture of pentane, hexane and heptane. Again the data from tables 3 and 4 is entered into the design tool to test the design feasibility. Then simulated to ensure accuracy.

<table>
<thead>
<tr>
<th>Component</th>
<th>Pentane</th>
<th>Hexane</th>
<th>Heptane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Composition</td>
<td>0.3</td>
<td>0.3</td>
<td>0.4</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>3.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.95</td>
<td>0.049</td>
<td></td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>0.05</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Component</th>
<th>Pentane</th>
<th>Hexane</th>
<th>Heptane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pentane</td>
<td>5.853654</td>
<td>4598.287</td>
<td>394.4148</td>
</tr>
<tr>
<td>Hexane</td>
<td>6.039243</td>
<td>5085.758</td>
<td>382.7941</td>
</tr>
<tr>
<td>Heptane</td>
<td>5.98627</td>
<td>5278.902</td>
<td>359.5259</td>
</tr>
</tbody>
</table>

Results:

The design results are feasible as observed in figure 8. The stripping and rectifying composition profiles generated by the feasibility design testing program fit the upper and lower ends of the profiles well to the graph produced by the HYSYS simulator once again as seen in figure 8. Again we see the same variations as in the first trial.
Our final ideal ternary trial will be methanol, isopropanol, and n-propanol with the specifications presented in tables 5 and 6. The same procedure is used as in the first two trials and the same results are hoped to be acquired.

**Ideal 3**

Table 5 Specifications Ternary Ideal 3

<table>
<thead>
<tr>
<th>Component</th>
<th>Methanol</th>
<th>Isopropanol</th>
<th>n-Propanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Composition</td>
<td>0.2</td>
<td>0.3</td>
<td>0.5</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>3.7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.96</td>
<td></td>
<td></td>
</tr>
<tr>
<td>bottoms mole fractions</td>
<td>0.02</td>
<td>0.36</td>
<td></td>
</tr>
</tbody>
</table>
Table 6 Antoine Coefficients Ternary Ideal 3

<p>| | | | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>7.513334</td>
<td>6468.101</td>
<td>396.2652</td>
</tr>
<tr>
<td>Isopropanol</td>
<td>7.180215</td>
<td>5596.813</td>
<td>327.2873</td>
</tr>
<tr>
<td>n-Propanol</td>
<td>6.683944</td>
<td>5414.961</td>
<td>303.9864</td>
</tr>
</tbody>
</table>

Results:

Once again these specifications are entered into the feasibility design testing program. Upon confirmation of feasibility, the data is then entered into HYSYS. And again we see the stripping and rectifying composition profiles generated by the feasibility design testing program again fit the upper and lower compositions of the column profiles and again the middle region is negligible for now. The results produced by the HYSYS simulator and design tool are seen in figure 9. The variations are still present in the third trial as well.

---

![Figure 9 Results for ideal ternary mixture 3](image-url)
3.2 Modeling of Vapor Pressures

As discussed earlier, there are variations in the Antoine equations used in the design and simulation of the column separations. The design feasibility testing program uses the Antoine equations presented in Henley and Seader, *Equilibrium-Stage Separation Operations in Chemical Engineering*, 1981. This equations is

\[ P_{\text{vap}} = P_c e^{\left( A - \frac{B}{T+C} \right)} \]  

(15)

and uses three coefficients, A, B, and C as well as the critical pressure to calculate vapor pressure. The temperature is in Fahrenheit and the critical pressure in psia.

The HYSYS simulator software uses the following Antoine equation

\[ P_{\text{vap}} = e^{\left( A + \frac{B}{T+C} + D\ln T + E T + F \right)} \]  

(16)

As can be seen in the above equation, HYSYS uses a much more extensive Antoine equations with coefficients A thru F to compute the vapor pressure. The temperature is in Kelvin the pressure in kilopascals.

A comparison was done between seven of the common components used in these trials to calculate the difference attained by these two equations. Using MathCAD to calculate the vapor pressures and convert them to the same units, these results were obtained as seen in Table 1.
Table 7 Pure Component Vapor Pressures

<table>
<thead>
<tr>
<th>Compound</th>
<th>Delphi PVap (kPa)</th>
<th>Hysys PVap (kPa)</th>
<th>Difference</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexane</td>
<td>53.61</td>
<td>52.753</td>
<td>0.857</td>
</tr>
<tr>
<td>Heptane</td>
<td>18.88</td>
<td>18.375</td>
<td>0.505</td>
</tr>
<tr>
<td>Octane</td>
<td>6.72</td>
<td>6.269</td>
<td>0.451</td>
</tr>
<tr>
<td>Methanol</td>
<td>55.39</td>
<td>55.731</td>
<td>0.341</td>
</tr>
<tr>
<td>Ethanol</td>
<td>29.78</td>
<td>29.627</td>
<td>0.153</td>
</tr>
<tr>
<td>Isopropanol</td>
<td>24.33</td>
<td>23.812</td>
<td>0.518</td>
</tr>
<tr>
<td>Propanol</td>
<td>12.25</td>
<td>12.156</td>
<td>0.094</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td></td>
<td>2.919</td>
</tr>
<tr>
<td>Average:</td>
<td></td>
<td></td>
<td>0.417</td>
</tr>
</tbody>
</table>

These differences in the Antoine equations contribute to the variations in the composition profiles.

Before presenting the non-ideal ternary mixtures trials a word must again be said about coefficients. This time the difference is Wilson coefficients. The HYSYS simulator, calculates the Wilson interaction coefficients by default. These coefficients can be entered manually as mentioned in the procedure section. Therefore, the Wilson coefficients obtained in Gmehling, Kolbe, et al. *Vapor-liquid equilibrium Data Collection, Vol. 1* Parts 1-6, 1983, are both entered into the design feasibility testing program as well as manually entered into the HYSYS simulator. By using these same coefficients the results are much closer between the two programs as will be seen in the trials.

### 3.3 Design and Analysis of Non-ideal Ternary Separations

**Non-ideal 1**

In the next three trials the focus will be the design and simulation of non-ideal ternary mixtures. The first design will be for a mixture of hexane, benzene, and toluene.
The data is entered as described in the procedure. The difference for this trials and the following ones will be that the Wilson activity coefficients must be entered manually into both the design tool and the simulator. The specifications are given in tables 8 and 9.

<table>
<thead>
<tr>
<th>Component</th>
<th>Hexane</th>
<th>Benzene</th>
<th>Toluene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Composition</td>
<td>0.3</td>
<td>0.3</td>
<td>0.4</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>5.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.518</td>
<td>0.48</td>
<td></td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>5e-7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 8 Specifications Ternary non-ideal 1

<table>
<thead>
<tr>
<th>Component</th>
<th>Hexane</th>
<th>Benzene</th>
<th>Toluene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexane</td>
<td>0</td>
<td>309.061</td>
<td>72.872</td>
</tr>
<tr>
<td>Benzene</td>
<td>757.712</td>
<td>0</td>
<td>268.239</td>
</tr>
<tr>
<td>Toluene</td>
<td>247.074</td>
<td>-189.605</td>
<td>0</td>
</tr>
</tbody>
</table>

Table 9 Wilson Interaction Coefficients Ternary non-ideal 1

Results:

The specifications have been entered into the feasibility testing program and proved feasible. This data is then entered into the HYSYS simulator and the results are displayed below as can be seen in figure 10. The composition profiles reflect each other in a very similar manner. Both the upper and lower ends of the column as well as the middle sections of the profiles fit very similarly. This can be attributed to the same Wilson coefficients used in both programs. The design tool does an accurate job of predicted both the feasibility and the compositions of the column sections in this case.
Non-ideal 2

Presented in trial 2 is a mixture of benzene, toluene, and p-xylene for separation. This mixture referred to BTX in industry, is common contaminant from chemical production. The mixture is first entered into the design feasibility tester as described in the previous trials using the specifications presented in tables 10 and 11. The results are shown below.

Table 10 Specifications non-ideal ternary 2

<table>
<thead>
<tr>
<th>Component</th>
<th>Benzene</th>
<th>Toluene</th>
<th>p-Xylene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Composition</td>
<td>0.3</td>
<td>0.3</td>
<td>0.4</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>2.25</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.99</td>
<td>0.0099</td>
<td></td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>1e-6</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 11 Wilson Interaction Coefficients non-ideal ternary 2

<table>
<thead>
<tr>
<th></th>
<th>Benzene</th>
<th>Toluene</th>
<th>p-Xylene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Benzene</td>
<td>0</td>
<td>-310.307</td>
<td>5.115</td>
</tr>
<tr>
<td>Toluene</td>
<td>323.122</td>
<td>0</td>
<td>118.693</td>
</tr>
<tr>
<td>p-Xylene</td>
<td>-0.04</td>
<td>-120.645</td>
<td>0</td>
</tr>
</tbody>
</table>

Figure 11 Results for non-ideal ternary mixture 2

Results:

The specifications have been entered into the feasibility testing program and proved feasible. This data is then entered into the HYSYS simulator and the results can be seen in figure 11 above. The upper and lower ends of the column compositions again fit well between the two programs as well as the liquid feed stage. The middle portions of the column profiles do not fit as well, but as in the ideal trials we are currently not interested in these parts for the purpose of these trials.

Non-ideal 3
And finally we again perform the same procedure on a mixture of methanol, ethanol, and n-propanol with the specifications listed in tables 12 and 13.

Table 12 Specifications non-ideal ternary 3

<table>
<thead>
<tr>
<th>Component</th>
<th>Methanol</th>
<th>Ethanol</th>
<th>n-Propanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Feed Composition</td>
<td>0.3</td>
<td>0.25</td>
<td>0.45</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>3.6</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.5499</td>
<td>0.4499</td>
<td></td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>5e-7</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 13 Wilson Interaction Coefficients non-ideal ternary 3

<table>
<thead>
<tr>
<th></th>
<th>Methanol</th>
<th>Ethanol</th>
<th>n-Propanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Methanol</td>
<td>0</td>
<td>-326.819</td>
<td>-987.209</td>
</tr>
<tr>
<td>Ethanol</td>
<td>323.122</td>
<td>0</td>
<td>3108.881</td>
</tr>
<tr>
<td>n-Propanol</td>
<td>1846.612</td>
<td>-829.156</td>
<td>0</td>
</tr>
</tbody>
</table>

Results:

The specifications have been entered into the feasibility testing program and proved feasible. As seen in above in figure 12, the compositions of the top and bottoms of the
column are very similar as expected as well as the middle stage compositions of the column, which are again neglected.

4 Ideal and Non-ideal Quaternary Trials

4.1 Quaternary Design Procedure

Before presenting the quaternary tests, we will first look at the four component design feasibility testing software. A screen shot of this program is presented in figure 13. The data is entered in exactly same method as with the ternary design, different only in that there is a fourth component for which data must be entered. There is only one choice of radio button specifications. Looking at the left side of the figure at the resulting profiles, the top plot is the stripping and rectifying profiles with component 1 on the x-axis and component 2 on the y-axis. The lower plot, is of the stripping and rectifying of component 4 versus component 1. The lower plot’s y-axis extents from zero to negative one, where in reality it is a positive one, and is presented in such a many for clarity and simplicity of the profile plots. A design is feasible if the rectifying and stripping profiles intersect on both plots and the intersection points of the two plots approximately align vertically.

4.2 HYSYS Simulation Procedure

The HYSYS simulations of quaternary mixtures are preformed in the same manner as stated in the procedure, except that instead of working with three components, one would choose four.
Again for ideal quaternary mixtures the same sources for Antoine coefficients are used for the vapor pressures. In the body of this paper there will be presented two ideal and one non-ideal quaternary mixture trials.

### 4.3 Design and Analysis of Ideal and Non-ideal Quaternary Separations

**Ideal 1**

For the first design calculation an ideal mixture of diethyl ether, hexane, heptane, and octane shall be used. The specifications from tables 14 and 15 will be entered into the design tool and simulator.
Table 14 Specifications Quaternary ideal 1

<table>
<thead>
<tr>
<th>Component</th>
<th>Diethyl Ether</th>
<th>Hexane</th>
<th>Heptane</th>
<th>Octane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>2.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.95</td>
<td></td>
<td></td>
<td>0.0001</td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td></td>
<td>0.00012</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 15 Antoine Coefficients Quaternary ideal 1

<table>
<thead>
<tr>
<th>Component</th>
<th>Diethyl Ether</th>
<th>Hexane</th>
<th>Heptane</th>
<th>Octane</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>5.976844</td>
<td>4614.723</td>
<td>388.8028</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6.039243</td>
<td>5085.758</td>
<td>382.1172</td>
<td></td>
</tr>
<tr>
<td></td>
<td>5.98627</td>
<td>5278.902</td>
<td>359.5259</td>
<td></td>
</tr>
<tr>
<td></td>
<td>6.4141</td>
<td>5947.491</td>
<td>360.26</td>
<td></td>
</tr>
</tbody>
</table>

Results:

As can be seen in the results in figure 14 on the below, the composition profiles of the quaternary mixture in the design feasibility test and the HYSYS simulation are similar in behavior, in that the upper and lower ends of the column compositions coincide once again. Again the variation of the difference of the profiles can be caused by the dissimilar Antoine coefficients used. Also as stated the design testing uses a continuous method of computing the profiles as HYSYS uses the tray by tray modeling.

Figure 14 Results for ideal quaternary mixture 1
**Ideal 2**

The next quaternary design separation will be an ideal mixture of diethyl ether, acetone, n-propanol, and n-butanol. Tables 16 and 17 give the specs needed to perform the design and simulation calculations.

### Table 16 Specifications Quaternary ideal 2

<table>
<thead>
<tr>
<th>Component</th>
<th>Diethyl Ether</th>
<th>Acetone</th>
<th>n-Propanol</th>
<th>n-Butanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>5.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.80</td>
<td></td>
<td>2e-8</td>
<td></td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>0.0000016</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### Table 17 Antoine Coefficients Quaternary ideal 2

<table>
<thead>
<tr>
<th>Component</th>
<th>Diethyl Ether</th>
<th>Acetone</th>
<th>n-Propanol</th>
<th>n-Butanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>Diethyl Ether</td>
<td>5.976844</td>
<td>4614.723</td>
<td>388.8028</td>
<td></td>
</tr>
<tr>
<td>Acetone</td>
<td>6.244412</td>
<td>5356.715</td>
<td>397.529</td>
<td></td>
</tr>
<tr>
<td>n-Propanol</td>
<td>6.683944</td>
<td>5414.961</td>
<td>303.9864</td>
<td></td>
</tr>
<tr>
<td>n-Butanol</td>
<td>6.303186</td>
<td>5225.324</td>
<td>274.4291</td>
<td></td>
</tr>
</tbody>
</table>

Results:

Observing the results in figure 15 on the following page, the composition profiles of the quaternary mixture in the design feasibility test and the HYSYS simulation are very close on the upper and lower ends of the column just as we hoped they would be. Again we see the variances in the plots of the composition profiles. Overall the results are again accurate in predicting feasibility of a design.
The final quaternary trial will be a non-ideal mixture of hexane, benzene, toluene and p-xylene. The specifications for given in tables 18 and 19. As in the non-ideal ternary trials the Wilson activity coefficients are again a factor.

Table 18 Specifications Quaternary non-ideal 1

<table>
<thead>
<tr>
<th>Component</th>
<th>Hexane</th>
<th>Benzene</th>
<th>Toluene</th>
<th>p-Xylene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>3.0</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.75</td>
<td></td>
<td></td>
<td>0.0001</td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>0.02</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 19 Wilson Interaction Coefficients Quaternary non-ideal 1

<table>
<thead>
<tr>
<th></th>
<th>Hexane</th>
<th>Benzene</th>
<th>Toluene</th>
<th>p-Xylene</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hexane</td>
<td>0</td>
<td>241.555</td>
<td>165.512</td>
<td>390.774</td>
</tr>
<tr>
<td>Benzene</td>
<td>94.169</td>
<td>0</td>
<td>-310.307</td>
<td>5.115</td>
</tr>
<tr>
<td>Toluene</td>
<td>83.646</td>
<td>323.122</td>
<td>0</td>
<td>118.693</td>
</tr>
<tr>
<td>p-Xylene</td>
<td>-164.704</td>
<td>-0.040</td>
<td>-120.645</td>
<td>0</td>
</tr>
</tbody>
</table>
Results:

Here in again in the non-ideal mixture, one sees the design composition profiles and the HYSYS composition profiles have matching compositions at the high and low ends of the column, see figure 16 below. These designs tested using the feasibility design software have proven to provide an accurate method for quickly designing a column separation that works without trial and error.

![Graph showing composition profiles](image)

**Figure 16 Results for non-ideal quaternary mixture 1**

**The Wilson Activity Coefficients coefficients of non-ideal Quaternary Design**

For both the ideal and non-ideal quaternary mixtures, hard copy data sources were not available for the retrieval of the Wilson interaction coefficients. The predicted HYSYS Wilson interaction coefficients were calculated and entered into the feasibility design program so that the programs will use the same numbers when finding the compositional profiles.
5 Column Sequencing

A mixture with three or more components may be separated into its pure components by using multiple columns. The distillate and bottoms from the first column are used as feed streams to two additional columns and so on.

A column sequence was run to test the feasibility program for a four component ideal mixture into its pure components. The we will be using the ideal mixture from the quaternary ideal mixture 1.

<table>
<thead>
<tr>
<th>Component</th>
<th>Diethyl Ether</th>
<th>Hexane</th>
<th>Heptane</th>
<th>Octane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
<td>0.25</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>2.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>0.95</td>
<td></td>
<td>0.0001</td>
<td></td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>0.00012</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The Antoine coefficients are the same as in table 15.

A flow chart of the sequence is shown in figure 17 below.
Column 1 separation results:

![Diagram showing separation results]

Figure 18 Results from first column in sequence

As can be seen in the results in figure 18, the composition profiles of the quaternary mixture in the design feasibility test and the HYSYS simulation are similar in behavior as illustrated in the earlier results for this separation.

Table 21 Feed Specifications for Column 3 in sequence

<table>
<thead>
<tr>
<th>Component</th>
<th>Diethyl Ether</th>
<th>Hexane</th>
<th>Heptane</th>
<th>Octane</th>
</tr>
</thead>
<tbody>
<tr>
<td>Composition</td>
<td>≈0</td>
<td>0.319</td>
<td>0.3405</td>
<td>0.3405</td>
</tr>
<tr>
<td>Reflux Ratio</td>
<td>2.5</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Distillate mole fractions</td>
<td>--</td>
<td>0.97</td>
<td>0.029</td>
<td>≈0</td>
</tr>
<tr>
<td>Bottoms mole fractions</td>
<td>--</td>
<td>0.002</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Results:

For the separation occurring in column #3 it has been treated as a ternary mixture since there is a negligible amount of diethyl ether in the bottoms from the first column. Thus
for the feasibility test is treated as an ideal ternary mixture. The results are presented in figure 19. Again the composition profiles reflect each other accurately.

![Figure 19 Column #3 Sequence results](image)

The design testing for column #2 and column #4 in the sequence could not be done seeing that the third component in each was so negligible that it had to be treated as a binary mixture, thus could not be designed using the feasibility test.
Conclusions

While distillation is a common practice in industry, there is still so many improvements that can be made in the design of distillation separations. This project has tested a more efficient method to designing feasible column separations. This method predicts whether a certain specifications/separations are possible given a certain component mixture. The program has proved to be both reliable, quick, and accurate in predicting the feasibility of a separation.

The trials performed were relevant in that they used both real component mixtures with both ideal and non-ideal interactions. Further improvements may be made to this feasibility testing program by making it apply to azeotropic mixtures as well as mixtures with more than four components. Small cosmetic changes can also be made to improve clarity such as labeling the axes on the resulting profile plots as well as profiles.

Personally, having never had a class on distillation, I have learned a lot about distillation and how it is applied in industry as well as the importance of having a robust and reliable design method in that is saves money and time. These ideas applied may be in industry to produce more efficient operations, recirculation of solvents, and an overall savings of time and money.
References


Tham, M.T. *Distillation, an Introduction*. 30 May 2003. 


Acknowledgements

- Dr. Libin Zhang- Post Doctorate
- Dr. Andreas Linniger- Advisor, LPPD director
- Dr. Christos Takoudis- NSF-REU director 2003
- The NSF for Financial support
Appendix A: Nomenclature

F- Feed
D- Distillate
B- Bottoms

r- reflux ratio
s- reboil ratio

C- number of components
n- stage number
h- height

\(x_{i,j}\) – liq. mole fraction of the \(i^{th}\) component from \(j^{th}\) stage

\(y_{i,j}\) - vap. mole fraction of the \(i^{th}\) component from \(j^{th}\) stage
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