Recovering Methanol through Distillation

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Recovering Methanol through Distillation: Characterizing Flooding Conditions and Correlation

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We have neither given nor received unauthorized aid in preparing this paper, nor have we concealed any violations of the Honor Code.

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ABSTRACT

Brown Industries, Inc. is attempting to scale-up the production of biodiesel from soybean oil. The company is investigating the use of a packed column to recover methanol from water so that it may be reused in the transesterification reaction step. The Rotation 2 team continued the work of Rotation 1 by trying to optimize the column’s operation. The column should perform optimally in the pre-flooding regime, which was thoroughly investigated.

Objectives included:

(a) Testing of reproducibility of Rotation 1’s control run
(b) Preparation of McCabe-Thiele plots for each of the experimental trials
(c) Develop a model that incorporates the packing mass transfer properties such that the column can be easily scaled up.
(d) Determination of pre-flooding operating boundary
(e) Assessment of flooding correlations

Investigation of the packed column was successful. Rotation 1’s control run was not found to be reproducible. McCabe-Thiele plots were prepared for each experimental trial and allowed for visualization of the mass balances and equilibrium interactions. A Python script model was created to calculate $H_{OG}$ for any given distillate and flow rate, and the $H_{OG}$ could be compared against vapor velocity to find the optimal operating conditions. A clear pre-flooding regime was identified for this column at a reboiler duty of 7600 W. The General Pressure Drop Correlation provided the best estimation of the column’s flooding behavior.
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INTRODUCTION

Brown Industries has decided to build a biodiesel plant using soybean oil as feedstock. The goal is to use the USDA Haas study [6] as a basis for a proposed plant layout. Within this layout, there are three streams where methanol can be recovered and recycled to the transesterification reactor. Using distillation, it is intended to recover methanol from these streams at a suitable purity for the transesterification reaction, as well as sufficiently purified water to be used in the POD extraction. It has been proposed that a new packing material from “Pro-Pak” may be more economical to use than an equivalent tray column.

On October 1, 2012, our team received a memo instructing us to execute a feasibility study of the distillation operation with water and methanol using a Pro-Pak packed column. Our assessment included pilot-scale studies that characterize the water-methanol distillation process, identify optimal operating conditions of the column, and describe flooding characteristics of the column.

Goals and Objectives

The following were the major objectives of our study:

- Perform the control run developed by Rotation 1 to assess the reproducibility of the product concentrations, the vapor and liquid flow rates, and the calculated mass-transfer parameters.
- Prepare a McCabe-Thiele diagram for each run
- Develop a model that incorporates the packing mass transfer properties such that the column can be easily scaled up.
- Determine the operating conditions that define the pre-flooding boundary, as well as if the manufacturer's flooding correlations or other generalized pressure drop correlation can be used to predict and prevent flooding in the column.

Key Results

The first objective for this rotation was to verify the control run from rotation 1. The experiment failed to reproduce a control run because rotation 1’s gas chromatography correlation was inaccurate, especially at the extremes. McCabe-Thiele assumptions were applied on the runs that were successfully produced, and graphs have been produced for each of them. The graphs were easily created from a model developed for packing mass transfer properties in the form of a Python programming language script. Other parameters were obtained from the Python script including vapor velocities, HOG and NOG values. The pre-flooding boundaries was determined to be at a reboiler duty (Q) equal to 7600 W, a vapor velocity in the stripping section of 64 m/min, and a vapor velocity in the rectifying section of 36 m/min for this specific column. The General Pressure Drop Correlation was found to be the most accurate in predicting flooding conditions in the column, especially when the column was allowed to reach steady state right before increasing the reboiler duty to reach flooding.
BACKGROUND

Theory of Distillation

The goal of the distillation process is to separate components from each other using differences in their volatilities—in this case, water and methanol. A distillation column is made up of several surfaces where each component can achieve equilibrium in the gas and liquid phase. Once equilibrium is reached on a stage, the gas phase (with the more volatile component now higher in concentration) travels upwards and the liquid phase (with the less volatile component now higher in concentration) travels downwards. For a new stage, the starting components are a combination of the above stage’s liquid phase and the below stage’s gas phase, which will then again reach equilibrium. Since the more volatile component will have a higher concentration in the gas phase again, it is intuitive that repeating this process iteratively will bring the more volatile component to the top of the column, and less volatile component to the bottom of the column. The top stream can be condensed, and the bottom stream can be reboiled to achieve relatively high purity product streams.

![Distillation Column Diagram](image)

**Figure 1:** Overview of distillation column function [5]

A trayed distillation column has multiple trays that provide surfaces for achieving equilibrium. In contrast, a packed column provides a continuous surface throughout the volume of the column. The efficacy of packing can be distilled down to two values: \( a \), the surface area per volume, and \( K \), which describes the mass transfer that occurs on that surface. Both these
values are combined into one term when reported, due to always appearing together in calculations. However, the mass transfer characteristics are different for each phase, so two values are used, \( K_x \), the mass transfer in the liquid phase, and \( K_y \), the mass transfer in the gas phase. \( K_x \) and \( K_y \) are functions of packing properties as well as operating conditions of the column, specifically vapor velocity.

During the operation of the column, the vapor velocity can be excessively high and prevent the liquid stream from the stage above from falling. This is called flooding, and is characterized by a sharp increase in column stage pressure and significant decrease in separation efficiency. Flooding is dependent on both liquid and gas velocities in the column, and it is extremely beneficial to know the operating limits of the column to minimize flooding.

**Control Run Reproducibility**

The control runs from this rotation could not be simply compared to the control runs from the previous rotation because there were changes in feed composition. When feed composition changes in a pinch region at the extreme purities, the composition of the product streams can be greatly affected. As such, in order to determine the reproducibility of this rotation’s data, the \( H_{OG} \) and the vapor velocity were calculated for each of the trials. If the data points fit onto the \( H_{OG} \) vs. vapor velocity correlation from Rotation 1, the runs were considered to be reproducible.

**Mass Balances – McCabe-Thiele Assumptions**

We needed to perform numerous calculations to reproduce the previous rotation’s work and to characterize the column for our experiments. This included mass balances in the different sections of the column. We assumed McCabe-Thiele conditions (constant-molar overflow) and used equations (1) and (2) to analyze the rectifying and the stripping sections of the column, respectively.

\[
y_r = \frac{L}{V} x_r + \frac{D}{V} x_D
\]  

where  
- \( L \) = Liquid flow through the rectifying section  
- \( V \) = Vapor flow through the rectifying section  
- \( D \) = Distillate flow  
- \( x_r \) = mole fraction of methanol in the rectifying section  
- \( x_D \) = mole fraction of methanol in the distillate  
- \( y_r \) = methanol mole fraction in rectifier vapor stream

\[
y_s = \frac{L'}{V'} x_s - \frac{B}{V'} x_B
\]  

where  
- \( L' \) = Liquid flow through the stripping section  
- \( V' \) = Vapor flow through the stripping section  
- \( x_s \) = mole fraction of methanol in the stripping section  
- \( x_B \) = mole fraction of methanol in the bottoms  
- \( y_s \) = methanol vapor fraction in stripper vapor stream
Vapor Velocity

The vapor velocity was varied by changing the reboiler duty, which changed the vapor flowrate into the column. The vapor velocity was calculated from equation (3).

\[ v = \frac{V}{A} \cdot \frac{1}{\rho_v} \]  

(3)

where 
- \( v \) = vapor velocity (superficial) 
- \( V \) = vapor molar flow rate 
- \( \rho_v \) = molar density of the vapor 
- \( A \) = cross-section area of the column

Mass Transfers Parameters

To find the mass-transfer parameters \( N_{OG} \) and \( H_{OG} \), we used equations (4) and (5) respectively.

\[ N_{OG} = \int_{y_1}^{y_2} \frac{dy}{y^*-y} \]  

(4)

where 
- \( N_{OG} \) = Number of mass transfer units 
- \( y^* \) = Methanol vapor fraction in equilibrium with liquid 
- \( y \) = vapor fraction

Equation (5) demonstrates how the height of the packing and the height of the transfer unit are related.

\[ H_{OG} = \frac{z}{N_{OG}} \]  

(5)

where 
- \( H_{OG} \) = Height of mass transfer unit (in) 
- \( z \) = the height of the section of the column (in)

\( H_{OG} \) was plotted against the vapor velocity, in order to determine the relationship between the two. This allowed us to find where \( H_{OG} \) is minimized and the column has the greatest throughput.

Flooding Characteristics

It was essential to identify the flooding limit of our column to operate the column at an optimal steady state condition. The Pro-Pak equipment manual has a flooding correlation that we used to analyze the limits of our column [4]. The correlation is demonstrated in equation (6):

\[ G = 270 \left(p_1\right)^{0.58} \left(p_2\right)^{0.42} \]  

(6)

where 
- \( G \) = mass vapor velocity at flooding (lb/ft^2/hr) 
- \( p_1 \) = density liquid phase (lb/ft^3) 
- \( p_2 \) = density of gas phase (lb/ft^3)

We also analyzed the flooding using a correlation that takes into consideration the pressure drop across the column. We used the Kister and Gill correlation, a generalized pressure drop
correlation (GPDC) to study the flooding in our experiment. Equation (7) demonstrates such correlation.

\[ \Delta P_{\text{Flood}} = 0.12 F_p^{0.7} \]  

(7)

where \( \Delta P_{\text{Flood}} \) = pressure drop across the column  
\( F_p \) = packing factor

The packing factor has been given to us in the Pro-Pak. The packing factor for the 0.24-inch protruded packing is 420. [4]

**MATERIALS AND METHODS**

The Brown Industries custom-built packed distillation column is depicted in Figure 2, below. Experimentation also required the use gas chromatography. The Brown Industries laboratory uses a Carle 100 gas chromatographer.

**Equipment and Materials**

The following equipment was used to perform the experiment:

- Feed tank reservoir (8-gallon stainless steel tank, a sight glass and safety filler cap)
- Reboiler (3.8-4.0 gallon cylindrical stainless steel tank)
- Boiler (covered with rubber insulation)
- Stainless steel sheathed bayonet-type 3-phase electric heater (15 Kw)
- Packed column (2 packed bed sections, 31 inches height)
- ¼-inch Pro-Pack stainless steel random packing
- Condenser (Pyrex and stainless steel shell-and-tube type heat exchanger)
- Distillate receiver (3-inch OD 12-inch long Pyrex glass tube)
- Instrumentation and control panel (voltmeter and ammeter for power input, boiler liquid level control, automatic operation switch, on/off switches for pumps and indicator lights)
- Thermocouples (17 type K thermocouples)
- Rotameters (measure feed, reflux and coolant flows)
- Turbine flow meter (measure feed, reflux and distillate and bottoms flow)
- Differential pressure gauge
- LabView software and computer

The materials and software used in experimentation are listed below:

- 2 mL sample vials for collection of gas chromatography samples
- 1 mL syringe with type 5 needle for sample extraction from the still
- 1 μL syringe for injection of 0.2 μL sample into chromatograph
- Microsoft Excel for data analysis and graph generation
- Python script for data analysis
- PeakSimple Software for GC data analysis
Figure 2: Schematic of the Brown Industries distillation apparatus [2]
Procedures

Initial Start-Up

1. Start up the column according to “Distillation Column Start-Up Procedure” in the equipment manual [2].
2. Set the column to the operating conditions for the control run: reflux ratio of 5, feed flow of 610 mL/min, and reboiler duty of 5000 W.
3. Allow column to achieve steady state operation (requires approximately 45 minutes).
4. Follow the “Guidelines to Steady State Determination,” below, to determine when the column is likely in steady state.
5. Once the column is believed to be in steady state, take samples from the feed, bottoms, and distillate ports according to “Gas Chromatograph Operating Instructions” in the equipment manual [2].
6. Analyze these samples by gas chromatography according to “Standard Procedure for the Analysis of Methanol in Water Mixtures” [1]. Use the calibration curves provided in this report to determine the methanol concentration based on the fractional area under the methanol and water peaks.
7. About 5 minutes later, take second distillate sample and analyze by gas chromatography. If the concentration is similar to the prior distillate sample, start a new trial in LabVIEW and proceed to the next trial. Else, repeat this step after another 5 minutes.

Subsequent Trials

1. Set the column to the new operating conditions (reflux ratio, feed flow, reboiler duty) according to the equipment manual [2].
2. Repeat steps 3-7 from above.

When experimentation is complete, shut down the column according to “Distillation Column Shut-Down Procedure” in the equipment manual [2].

Guidelines to Steady State Determination

Manual calculations and LabVIEW trends can be used to confirm that the column is at steady state conditions. LabVIEW can be used to ensure that there are no major fluctuations in the temperature profile, pressure profile, and flow rates. It is particularly important that these values are not trending upward or downward. Manual mass and energy balances can also be used to verify steady state. (Accumulation should be zero.) The final method for ensuring steady state conditions is testing the methanol content in the feed and product streams. There are detailed steps in the procedure based on this criterion.

Trials

The controllable parameters on this apparatus are the feed rate, reboiler duty, and reflux ratio. An additional disturbance variable is the concentration of methanol in the feed, which is unfortunately variable throughout the day. The reflux ratio and reboiler duty were varied in this rotation’s experimentation in order to determine optimal operating conditions for the column (i.e. – those in which HOG was minimized so that more separation is performed per packing height).
A summary of experimental trials that were allowed to reach steady state is provided in Table 1, below.

**Table 1: Steady-state experimental trials**

<table>
<thead>
<tr>
<th>Trial</th>
<th>Reflux ratio</th>
<th>Reboiler duty (W)</th>
<th>Feed flow (mL/min)</th>
<th>Feed conc. (mol%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-1 (C)</td>
<td>5</td>
<td>5000</td>
<td>610</td>
<td>2.55%</td>
</tr>
<tr>
<td>2-8</td>
<td>7</td>
<td>5000</td>
<td>610</td>
<td>2.82%</td>
</tr>
<tr>
<td>3-1 (C)</td>
<td>5</td>
<td>5000</td>
<td>610</td>
<td>2.45%</td>
</tr>
<tr>
<td>3-2</td>
<td>5</td>
<td>6900</td>
<td>610</td>
<td>3.31%</td>
</tr>
<tr>
<td>3-3</td>
<td>5</td>
<td>7250</td>
<td>610</td>
<td>3.16%</td>
</tr>
<tr>
<td>3-4</td>
<td>5</td>
<td>7600</td>
<td>610</td>
<td>3.18%</td>
</tr>
<tr>
<td>3-5</td>
<td>5</td>
<td>7825</td>
<td>610</td>
<td>2.65%</td>
</tr>
</tbody>
</table>

(C) indicates control run conditions

A number of trials were also run where the column was not allowed to reach steady state operation. In these trials, distillate samples were collected. A summary of the unsteady state experimental trials is provided in Table 2, below.

**Table 2: Unsteady-state experimental trials**

<table>
<thead>
<tr>
<th>Trial</th>
<th>Reflux ratio</th>
<th>Reboiler duty (W)</th>
<th>Feed flow (mL/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2-2</td>
<td>5</td>
<td>5550</td>
<td>610</td>
</tr>
<tr>
<td>2-3</td>
<td>5</td>
<td>6125</td>
<td>610</td>
</tr>
<tr>
<td>2-4</td>
<td>5</td>
<td>6675</td>
<td>610</td>
</tr>
<tr>
<td>2-5</td>
<td>5</td>
<td>7250</td>
<td>610</td>
</tr>
<tr>
<td>2-6</td>
<td>5</td>
<td>7825</td>
<td>610</td>
</tr>
<tr>
<td>2-7</td>
<td>5</td>
<td>8375</td>
<td>610</td>
</tr>
<tr>
<td>2-9</td>
<td>7</td>
<td>6125</td>
<td>610</td>
</tr>
<tr>
<td>2-10</td>
<td>7</td>
<td>7250</td>
<td>610</td>
</tr>
<tr>
<td>2-11</td>
<td>7</td>
<td>8375 → 7700</td>
<td>610</td>
</tr>
</tbody>
</table>

**RESULTS AND ANALYSIS**

**Reboiler Duty Correlation**

In order to understand the heating duty delivered by the reboiler, a correlation between the reboiler setting (% of power) and reboiler duty was generated for the range of typical working powers. This correlation should prove useful during future experimentation with the distillation column.

In order to develop the correlation, the reboiler duty was measured at different settings. The reboiler duty was indirectly measured by reading from the electricity meter on the reboiler the amount of energy that was consumed in a given time interval (60 s). Reboiler duty was calculated as:

\[ P = \frac{\Delta E_m}{\Delta t} \]  

(8)
where

\[ P = \text{Reboiler duty (W)} \]
\[ \Delta E_m = \text{Electricity meter energy registered (W-h)} \]
\[ \Delta t = \text{Time elapsed (60 s = 0.01667 h)} \]

The data was plotted, and a strong linear correlation was found. (See Figure 3, below.)

![Figure 3: There is a strong linear correlation between the reboiler power setting (% of power) and the measured reboiler duty (W).](image)

The correlation, which has an \( R^2 \) value of 0.9974, is defined in Equation 9:

\[
P = 113.53r - 468.62
\]  

(9)

where \( r = \text{Reboiler power setting (% of power)} \)

The residuals for the correlation, shown in Figure 4, below indicate that the correlation becomes less accurate at higher reboiler duties, but the error is still within about 1%. The correlation was tested on later lab dates, and the correlation still described the relationship between the reboiler setting and duty.
Figure 4: The reboiler duty correlation residuals have a “cornucopia” shape indicating increasing error at higher reboiler duties.

Gas Chromatography Recalibration

Rotation 1 provided a calibration curve for determining the concentration of methanol in experimental samples by gas chromatography. They took gas chromatography measurements of a set of samples with known methanol content and reported how the fraction of the integrated methanol peak area correlated to the weight fraction of methanol in the standard. While this correlation provided a good baseline, it did not take into consideration the physical limits that 0 methanol peak area should correspond to a pure water sample and that 0 water peak area should correspond to a pure methanol sample. This oversight led to large error when calculating methanol concentrations in the product streams.

Rotation 1’s data was used to generate a new piecewise-defined gas chromatography calibration curve that was more accurate at the extremes of sample purity. For samples with low methanol concentration, a correlation was made using only the chromatography data from the low-methanol concentration samples, and the fit was forced through the origin. (See Figure 5, below.) For samples with high methanol concentrations, a correlation was constructed using only the chromatography data from the low-water concentration samples, and the methanol concentration was plotted against the water peak relative area. (This method is more accurate because gas chromatography measurement errors are less exaggerated when the peaks are smaller.) Further, the second correlation was forced to show pure methanol when there was no water peak. (See Figure 6, below.) A gas chromatography correlation was not developed for intermediate methanol concentrations as there was no need for such during this rotation.
The correlation for low methanol concentrations (less than 25 wt%) has an $R^2$ value of 0.998 and follows the equation:

$$x_{MeOH} = 0.9397A_{MeOH}$$  \hfill (10)

where $x_{MeOH}$ = Methanol weight percent
$A_{MeOH}$ = Methanol relative peak area (%)

The correlation for high methanol concentrations (greater than 95 wt%) has an $R^2$ value of 0.729 and follows the equation:

$$x_{MeOH} = 100 - 0.4561A_{H2O}$$  \hfill (11)

where $A_{H2O}$ = Water relative peak area (%)
Determination of Flooding

In order to determine where flooding occurred during the experimental trials, the column pressure drop was plotted against the reboiler duty. There was a neat linear trend when the column was not flooding, followed by a spike in the slope when the column began to flood, as depicted in Figure 7, below.

This approach allowed for pinpointing of which trial was nearest to flooding conditions. For instance, at R=7, it is clear that the column began to flood at reboiler duty of about 7600 W, which corresponds to Trial 2-11. A vapor velocity could be calculated (using the script described in Appendix C) for this trial to estimate the approximate flooding velocity.

Flooding Correlations

We analyzed our data to determine which of the two flooding correlations—Pro-Pak and the GPDC—better reflects the packing’s actual behavior. We found that the GPDC gives a close approximation to flooding when the column is allowed time to reach steady state. The experimental pressure drop when flooding at a reflux ratio of 5 was 8.5 in-H₂O (trial 3-6), while the GPDC predicted value was 8.23 in-H₂O (3.17% error).

The GPDC predicted the flooding pressure drop poorly when the column was not given enough time to reach steady state. For instance, at the same reflux ratio (5), the column flooded with a pressure drop of 10 in-H₂O (error of at least 17.70%). When the column flooded with a reflux ratio of 7, the pressure drop was 11.6 in- H₂O (error of 29.05%). The second and third columns of Table 3 represent the findings on the GPDC and the experimental values that compare to it. Appendix B shows all the calculations that were performed to obtain the flooding correlation information.
The Pro-Pak correlation does not predict the flooding parameters nearly as well. In the rectifying section, the experimental mass vapor velocity at the flooding point was 408 lb/ft²-h, while the Pro-Pak correlation predicted 770.02 lb/ft²-h (error of 88.73%). The last two columns in Table 3 show the mismatch between the Pro-Pak correlation and experimental flooding data. More details can be found in Appendix B.

Table 3. Flooding Correlation vs. Observed Flooding

<table>
<thead>
<tr>
<th>Trial</th>
<th>Rectifying Section</th>
<th>Stripping Section</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Experimental Pressure Drop (in-H₂O)</td>
<td>GPDC Pressure Drop (in of H₂O)</td>
</tr>
<tr>
<td></td>
<td>3-6 (R=5)</td>
<td>8.50</td>
</tr>
<tr>
<td></td>
<td>2-7 (R=5)</td>
<td>10.00</td>
</tr>
<tr>
<td></td>
<td>2-11 (R=7)</td>
<td>11.60</td>
</tr>
<tr>
<td></td>
<td>3-6 (R=5)</td>
<td>8.50</td>
</tr>
<tr>
<td></td>
<td>2-7 (R=5)</td>
<td>10.00</td>
</tr>
<tr>
<td></td>
<td>2-11 (R=7)</td>
<td>11.60</td>
</tr>
</tbody>
</table>

**H₀G vs. Vapor Velocity Correlations**

The script described in Appendix C follows procedures and equations outlined by Lyon et al. [3] to calculate vapor velocities and the H₀G mass transfer coefficient, as well as to produce McCabe-Thiele plots (which are available in Appendix D). When the H₀G is plotted against the vapor velocity in Figures 8 and 9, the limit of vapor velocity when flooding is approached becomes evident. Once the reboiler duty is increased enough to induce flooding, the mass transfer becomes less efficient, and H₀G quickly increases. In both the stripper and in the rectifier, H₀G appears to be minimized at vapor velocities just below the flooding regime.
Figure 8: The plot of $H_{OG}$ against vapor velocity in the rectifying section shows that the vapor velocity approaches a maximum as the column approaches the flooding regime.

Figure 9: The plot of $H_{OG}$ against vapor velocity in the stripping section shows that the vapor velocity approaches a maximum as the column approaches the flooding regime.

**DISCUSSION**

While operating the distillation column, many opportunities for error arose. One source was that we were unable to compare our data to Rotation 1’s due to an incorrect correlation at extreme
concentrations. The very purpose of our lab was to determine the flooding limits of the reactor, meaning we were operating at extremes, and hence had to revise the correlation.

We also noticed that the closed loop of the column led to changing feed concentration over time. During our analysis, small variations of the feed concentration led to large swings on the product stream composition due to proximity to a pinch point. This error can be slightly minimized by taking multiple samples, although a more permanent solution would be to add an impeller to the storage tank, furthering mixing.

Also, we noticed during our calculations that they required the molar volume of the vapor. In the column, due to changing temperature and/or concentration, the molar volume is not constant, although there is only a 7% difference between each extreme. We took Rotation 1’s values of 36.29 mol/m³ for the rectifying section and 33.59 mol mol/m³ for the stripping section.

For the Pro-Pack Model, the source of the almost 400% error was unclear. Manipulating the data, it is apparent that reducing the vapor and liquid densities to 25% of their original values was the only way to get near the experimental vapor mass velocity. While we have explored some possibilities for this error, such as temperature and pressure sensitivity, we could not find a valid explanation for this large error.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions
The first objective for this rotation was to verify the control run from Rotation 1. The experiment failed to reproduce a control run because Rotation 1’s gas chromatography correlation was inaccurate, especially at the extremes. McCabe-Thiele assumptions were applied on the runs that were successfully produced, and graphs have been produced for each of them. The graphs were easily created from a model developed for packing mass transfer properties in the form of a Python programming language script. Other parameters were obtained from the Python script including vapor velocities, HOG and NOG values. The pre-flooding boundaries were determined to be at a reboiler duty (Q) equal to 7600 W, a vapor velocity in the stripping section of 64 m/min, and a vapor velocity in the rectifying section of 36 m/min for this specific column. The General Pressure Drop Correlation was found to be the most accurate in predicting flooding conditions in the column, especially when the column was allowed to reach steady state right before increasing the reboiler duty to reach flooding. Adding a mixer to the feed tank would greatly help reduce the fluctuations in the feed compositions. Improvements to the gas chromatography SOP are necessary to reduce error in the future works using chromatography in Brown Industries.

Recommendations
The consistency of feed composition was always an issue when we ran our experiments. The column is fed from an 8-gallon tank that is continuously replenished by the distillate and bottoms product streams. It has been noted by both the first and second rotations that the methanol composition of the feed can vary up to 2 mol% throughout a 4-hour lab session. When the feed is near a pinch point (in the low methanol concentration regime), these fluctuations can have a major impact on the compositions of the product streams. To make the feed more uniform, we recommend that a mixer be installed in the tank, allowing the contents to be well-mixed before
being fed back to the column. The mixing of the feed tank should reduce fluctuations in feed composition, allowing for more consistent results.

The gas chromatography standard operating procedure did not give enough details in certain steps. The SOP does not emphasize the importance of cleaning the syringe of any previous residues and fluids. We recommend that the user pump the syringe about 10 times (as opposed to 2) before finally taking a sample from the apparatus. We also recommend that the user apply the same method while taking the 0.2µL samples before introducing them into the gas chromatographer. The user must also make sure that the tip of the needle is bubble-free. The needle must be carefully inserted once the sample is collected, and the user should push the plunger swiftly so that the micro-sample is properly and deeply inserted into the gas chromatographer.

The gas chromatography SOP should also include details about fixing and re-integrating the boundaries on PeakSimple software. The readings were sometimes misaligned, and the areas calculated did not truly reflect the fraction of the components. The area could be adjusted by moving the ends of the red horizontal interval left or right in a way that would include the vast majority of the peak that it is supposed to encompass. The user should click on Edit, then re-integrate, so the software calculates the new areas.
REFERENCES


<table>
<thead>
<tr>
<th>Term</th>
<th>Definitions</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>B</td>
<td>Bottoms flow</td>
<td>mole/min</td>
</tr>
<tr>
<td>D</td>
<td>Distillate flow</td>
<td>mole/min</td>
</tr>
<tr>
<td>F</td>
<td>Feed flow</td>
<td>mole/min</td>
</tr>
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<td>F_L</td>
<td>Liquid fraction of feed (after flash)</td>
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<tr>
<td>F_V</td>
<td>Vapor fraction of feed (after flash)</td>
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<td>Hog_r</td>
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<td>in</td>
</tr>
<tr>
<td>Hog_s</td>
<td>Height of a gas-phase mass-transfer unit in the stripper</td>
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</tr>
<tr>
<td>L</td>
<td>Liquid flow in the rectifier</td>
<td>mole/min</td>
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<tr>
<td>L'</td>
<td>Liquid flow in the stripper</td>
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<td>-</td>
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<tr>
<td>Nogs</td>
<td>Number of transfer units in the stripper</td>
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<tr>
<td>q</td>
<td>Feed quality</td>
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<tr>
<td>V</td>
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</tr>
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<td>V'</td>
<td>Vapor flow in the stripper</td>
<td>mole/min</td>
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<tr>
<td>V'L</td>
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</tr>
<tr>
<td>x'_1</td>
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<td>x_D</td>
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<td>x_F</td>
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<tr>
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<td>Watts</td>
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<td>hours</td>
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<tr>
<td>$r$</td>
<td>Reboiler power setting</td>
<td>% of power</td>
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<tr>
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### Appendix B: Flooding Correlation Data

**Rectifier**

| Packing Factor | 420 |

<table>
<thead>
<tr>
<th><strong>Experimental Pressure Drop</strong></th>
<th><strong>GPDC Pressure Drop</strong></th>
<th><strong>Temp Thermocouple 7 Celcius</strong></th>
<th><strong>Trial</strong></th>
<th><strong>Density water (lb/ft^3)</strong></th>
<th><strong>Density Methanol (lb/ft^3)</strong></th>
<th><strong>X_methanol</strong></th>
<th><strong>Liquid Density</strong></th>
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<tr>
<th><strong>Trial</strong></th>
<th><strong>Gas Density (mol/m^3)</strong></th>
<th><strong>mol% methanol</strong></th>
<th><strong>Methanol gas density (g/m3)</strong></th>
<th><strong>H2O gas density (g/m3)</strong></th>
<th><strong>Methanol gas density (lb/ft^3)</strong></th>
<th><strong>H2O vapor density (lb/ft^3)</strong></th>
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<tr>
<th><strong>Trial</strong></th>
<th><strong>Pro-Pak mass vapor velocity at flooding</strong></th>
<th><strong>Experimental vapor velocity (m/min)</strong></th>
<th><strong>Liquid Density (ft/lb^3)</strong></th>
<th><strong>Gas Density</strong></th>
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B-1
## Stripping

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<th>Trial</th>
<th>Temp at thermocouple</th>
<th>Temp Bottoms</th>
<th>Density water</th>
<th>Density Methanol</th>
<th>Methanol gas density</th>
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<th>H2O gas density (g/m³)</th>
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<th>H2O gas density (lb/ft³)</th>
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<th>Experimental vapor velocity (m/min)</th>
<th>ft/hr</th>
<th>Liquid Density (ft/ft³)</th>
<th>Gas Density (lb/ft³)</th>
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Appendix C: Vapor Velocity, \( H_{OG} \)-\( N_{OG} \) Calculator, McCabe-Thiele Script

The script for calculating \( H_{OG} \) and vapor velocity was written in Python. The script may be reused by Rotation 3 for quick processing of output files from LabVIEW to perform their necessary calculations. The code is commented so that the flow of logic may be more easily followed. The code follows the data analysis procedure set forth by Lyon et al in Rotation 1.

```python
import Tkinter, tkFileDialog, re, csv

from pylab import *

#Constants
density_meoh = .792
density_water = 1
mw_meoh = 32.04
mw_water = 18.02
Cp = 4.186 #J/g K
d_Hvap = 40.65 # KJ / mol

#Additional Functions
def find_nearest(array,value):
    idx=(np.abs(array-value)).argmin()
    return idx

def vol_mol(flow,molfrac_meoh):
    flow_mol = flow * (molfrac_meoh * density_meoh / mw_meoh + (1-
molfrac_meoh) * density_water / mw_water)
    return flow_mol

def mol_weight(molfrac_meoh):
    mw = molfrac_meoh * mw_meoh + (1-molfrac_meoh) *
mw_water
    return mw

def intersection(m1,b1,m2,b2):
```

C-1
\[ x = \frac{(b_2-b_1)}{\text{float}(m_1-m_2)} \]
\[ y = m_1 \times x + b_1 \]
return \([x,y]\)

def my_range(start, end, step):
    while start <= end:
        yield start
        start += step

# take away pop up window
root = Tkinter.Tk()
root.withdraw()

# open file dialogue
filesa = tkFileDialog.askopenfilename(title='Pick all trial files')
# only need following line on windows
files = re.findall('\{(.*?)}', "".join(filesa))
files = filesa
# get list of all file names to process
params = []
definition = ['reboiler duty kW', 'Reflux Ratio', 'Feed MeOH mol',
'Distillate', 'Bottoms']
# for x in xrange(5):
#    params.append(input(definition[x]+': '))

openfile = open(files, 'rb')
# skip first twenty lines
for null in xrange(0,22):
    next(openfile)
trial_file = csv.reader(openfile, delimiter=' ')
headers = ['Blank', 'Time', 'Temp0', 'Temp1', 'Temp2', 'Temp3', 'Temp4',
'Feed_Flow', 'Reflux_Flow', 'Bottoms_Flow', 'DeltaP']
for header in headers:
    exec(header + ' = []')

alldata = [Blank, Time, Temp0, Temp1, Temp2, Temp3, Temp4, Temp5, Temp6,
           Temp7, Temp8, Temp9, Temp10, Temp11, Temp12, Temp13, Temp14, Temp15, Temp16,
           DoverL, Distillate_Flow, Feed_Flow, Reflux_Flow, Bottoms_Flow, DeltaP]

for line in trial_file:
    for x in xrange(0,len(headers)):
        alldata[x].append(line[x])

alldata.pop(0)
alldata.pop(0)
alldata = [[float(float(j)) for j in i] for i in alldata]

finalvalues = []

for x,header in enumerate(alldata):
    finalvalues.append(sum(header)/len(header))

headers.pop(0)
headers.pop(0)

finaldict = dict(zip(headers, finalvalues))

gcdata_all = []
gcdata_all.append({‘Trial’ : ‘2-1’, ‘Reflux_Ratio’ : 5. , ‘x_f’ : .0255, ‘x_d’ : .8652, ‘x_b’ : .0097})
gcdata_all.append({‘Trial’ : ‘3-1’, ‘Reflux_Ratio’ : 5. , ‘x_f’ : .024948, ‘x_d’ : .901351, ‘x_b’ : .009798})
gcdata_all.append({‘Trial’ : ‘3-2’, ‘Reflux_Ratio’ : 5. , ‘x_f’ : .033184, ‘x_d’ : .904852, ‘x_b’ : .001641})
gcdata_all.append({‘Trial’ : ‘3-3’, ‘Reflux_Ratio’ : 5. , ‘x_f’ : .031629, ‘x_d’ : .89245, ‘x_b’ : .001349})
gcdata_all.append({‘Trial’ : ‘3-4’, ‘Reflux_Ratio’ : 5. , ‘x_f’ : .031795, ‘x_d’ : .881089, ‘x_b’ : .00118})
gcdata_all.append({‘Trial’ : ‘3-5’, ‘Reflux_Ratio’ : 5. , ‘x_f’ : .026543, ‘x_d’ : .867892, ‘x_b’ : .000995})
gcdata = gcdata_all[6]
r = matplotlib.mlab.csv2rec('yxcsv.csv')

d1 = plt.figure()
plt.axis([0, 1, 0, 1])
plt.suptitle('McCabe-Thiele Plot - Trial ' + gcdata.get('Trial'))
plt.xlabel('liquid mol % ' + 'MeOH')
plt.ylabel('vapor mol % ' + 'MeOH')
plt.grid(True)

plt.plot(r.x,r.y, figure = d1)
plt.plot(r.x,r.x, figure = d1)

d1.canvas.draw()
show()

#Draw Rectifying Line

#Find m and b for rectifying line
rect_slope = gcdata.get('Reflux_Ratio')/(gcdata.get('Reflux_Ratio')+1)
rect_intercept = gcdata.get('x_d')*(1-rect_slope)

#Draw Feed Line
plt.plot([gcdata.get('x_f'),gcdata.get('x_f')], [0,gcdata.get('x_f')], figure = d1)

d1.canvas.draw()

#find m and b for q line
#Temp11 is feed, Temp0 is bottoms
q = (d_Hvap + mol_weight(gcdata.get('x_f')) * (1./1000) * Cp *
(finaldict.get('Temp0')-finaldict.get('Temp11')))/d_Hvap
feed_slope = q/(q-1)
feed_intercept = gcdata.get('x_f')*(1-feed_slope)

#Find intersection point
feed_intersection =
intersection(feed_slope, feed_intercept, rect_slope, rect_intercept)

#Draw q-line
plt.plot([gcdata.get('x_f'), feed_intersection[0]], [gcdata.get('x_f'), feed_intersection[1]], figure = d1)
d1.canvas.draw()
show()

#Stripping Section

#section slope
strip_slope = (feed_intersection[1] - gcdata.get('x_b'))/float(feed_intersection[0] - gcdata.get('x_b'))
strip_intercept = gcdata.get('x_b')*(1-strip_slope)

#draw strip line
plt.plot([gcdata.get('x_b'), feed_intersection[0]], [gcdata.get('x_b'), feed_intersection[1]], figure = d1)
d1.canvas.draw()
show()

#Draw Rectifying Line
plt.plot([gcdata.get('x_d'), feed_intersection[0]], [gcdata.get('x_d'), feed_intersection[1]], figure = d1)
d1.canvas.draw()
show()

#Vapor Velocities

#Stripping Section
Vb = 1/float(strip_slope-1)
Bottoms_Flow_mol = vol_mol(finaldict.get('Bottoms_Flow'), gcdata.get('x_b'))
Vapors_Flow_mol = Bottoms_Flow_mol*Vb
Vapor_Velocity_Strip = Vapors_Flow_mol/33.59/0.004560367

#Rectifying Section
Distillate_Flow_mol = vol_mol(finaldict.get('Distillate_Flow'), gcdata.get('x_d'))
Vapor_Flow_mol = Distillate_Flow_mol*(gcdata.get('Reflux_Ratio')+1)
# vol/mol calculated from average, and small number is cross-sectional area of
column - superficial veloicty
Vapor_Velocity_Rect = Vapor_Flow_mol/36.29/0.004560367

#Integrate
upper_bound_index = find_nearest(r.y, gcdata.get('x_d'))
lower_bound_index = find_nearest(r.y, feed_intersection[1])
rect_line = []
strip_line = []
for x in r.x:
    rect_line.append(rect_slope * x + rect_intercept)
    strip_line.append(strip_slope * x + strip_intercept)
sum = 0
for x in xrange(lower_bound_index, upper_bound_index):
    sum = sum + 1/float(((r.y[x] - rect_intercept)/float(rect_slope) - r.x[x]))*(r.y[x+1]-r.y[x])
Nog_rect = sum
Hog_rect = 18./Nog_rect

#Rectifying Section
upper_bound_index = find_nearest(r.y, feed_intersection[1])
lower_bound_index = find_nearest(r.y, gcdata.get('x_b'))
sum = 0
for x in xrange(lower_bound_index, upper_bound_index):
    sum = sum + 1/float(((r.y[x] - strip_intercept)/float(strip_slope) - r.x[x]))*(r.y[x+1]-r.y[x])

Nog_strip = sum
Hog_strip = 12./Nog_strip

d1.savefig(files + 'PICTURE.png')

Reflux_Ratio = gcdata.get('Reflux_Ratio')
x_f = gcdata.get('x_f')
x_d = gcdata.get('x_d')
x_b = gcdata.get('x_b')

allvalues = [files, rect_slope, rect_intercept, x_d, feed_slope,
feed_intercept, x_f, feed_intersection[0], feed_intersection[1], strip_slope,
strip_intercept, x_b, Vapor_Velocity_Strip, Vapor_Velocity_Rect, Nog_strip,
Hog_strip, Nog_rect, Hog_rect]

allvaluestext = [files', 'rect_slope', 'rect_intercept', 'x_d', 'feed_slope',
'feed_intercept', 'x_f', 'feed_intersection[0]', 'feed_intersection[1]',
'strip_slope', 'strip_intercept', 'x_b', 'Vapor_Velocity_Strip',
'Vapor_Velocity_Rect', 'Nog_strip', 'Hog_strip', 'Nog_rect', 'Hog_rect']

finalvaldict = dict(zip(allvaluestext, allvalues))

with open(files + 'DATA.csv','wb') as f: #Remember `newline="\n"` in Python 3.x
    w = csv.DictWriter(f, finalvaldict.keys())
    w.writeheader()
    w.writerow(finalvaldict)
Appendix D: McCabe-Thiele Plots and Data

Figure D.1: R=5, Q=5000W
Figure D.2: $R=7$, $Q=5000W$
Figure D.3: R=5, Q=5000W
Figure D.4: R=5, Q=6900W
Figure D.5: R=5. Q=7250W
Figure D.6: $R=5$, $Q=7600$ W
Figure D.7: R=5, Q=7825W
### GC Data

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<th>Trial #</th>
<th>Q</th>
<th>R</th>
<th>x₁</th>
<th>x₃</th>
<th>x₅</th>
<th>Nog - Rect</th>
<th>Hog - Rect</th>
<th>Nog - Strip</th>
<th>Hog - Strip</th>
<th>Rectifying Section</th>
<th>Stripping Section</th>
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<td>0.925</td>
<td>0.011</td>
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### Feed Line

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<th>Slope</th>
<th>Intercept</th>
<th>x</th>
<th>y</th>
<th>Slope</th>
<th>Intercept</th>
<th>Slope</th>
<th>Intercept</th>
<th>Vb</th>
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<td>0.833</td>
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</table>

**Figure D.8:** Data Table for Trials