

Group 8

Batch Distillation

Lab 6

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Executive Summary

Our goal in this experiment is to understand batch distillation. Then, perform a McCabe Thiele analysis and apply the Rayleigh equation to characterize the distillation column. We will operate a three-trayed column under constant reflux policy. We will separate Acetone from Butanol. The Initial mixture is at 19.6% Acetone with the rest being Butanol. The reflux ratio is estimated, and an equilibrium relationship was established based off relative volatility, which is assumed to be constant. Finally, the column is to be scaled, if applicable, based off of our data and analysis.

From our data, we establish a reflux ratio of 15.9 ± 0.253 . Relative volatility was estimated to be 2.42 ± 1.13 , which agrees with literature values. After solving for the right- and left-hand side, the Rayleigh equation did close but only due to the large error in the right-hand side. Therefore, it is questionable whether it actually closes or not. Reasons for the Rayleigh equation not closing may be due to possibly invalid assumptions of constant densities or perhaps due to the constant removal of liquids for GC analysis.

Generally, the errors can be attributed to the collected total reflux data, which are inconsistent with expectations since the total reflux acetone concentrations do not adhere to a supposed downward trend. Due to the fact that our data heavily depends on the total reflux data, it is expected to be flawed.

Without accurate total reflux data and other information, the design question was deemed undoable as well as unrealistic (see Design Question Details). A scale-up could be possible if the experiment were to be redone such that the total reflux is reached. Moreover, redoing the experiment with the same initial concentrations as described in the design question will help relate the experiment to the scale-up better. In general, to obtain better results for future experiments, more time should be allotted to obtain total reflux before the valve is opened and the timer is started.

Table of Contents

Executive Summary	1
List of Figures	3
List of Tables.....	3
Objectives.....	4
Introduction	4
Theory	4
Development of Rayleigh Equation	5
Constant Reflux – Average Distillate Composition and Batch Time	7
Constant Distillate Composition – Batch Time and Final Batch Quantity	8
McCabe Thiele Graphical Analysis.....	8
Relative Volatility	10
Integration of Rayleigh Equation and Incorporation of Relative Volatility	11
Boil up Calculation.....	12
Results/Data	13
Analysis.....	15
Conclusions	17
References.....	17
Appendices.....	18
Sample Calculations	18
Error Sample Calculations	20
Error Analysis	25
Design Questions Details.....	26
Raw Data	26
Experimental Procedure.....	29
Laboratory Safety Analysis	30
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List of Figures

Figure 1: Simple Batch Distillation Apparatus [1]	5
Figure 2: Constant Reflux at multiple times [2]	7
Figure 3: Rectifying Section Mass Balance Analysis [2]	9
Figure 4: Rectifying Section McCabe Thiele Diagram [2]	10
Figure 5: Relative Volatility Effect on Equilibrium Curve [2]	11
Figure 6: Acetone concentration over time	13
Figure 7: n-Butanol concentration over time	14
Figure 8: x-y graph of experimental acetone/n-butanol system	14
Figure 9: $1/(x_D - x_B)$ vs x_B graph for integrating RHS of Rayleigh equation	15

List of Tables

Table 1: Key batch distillation values and associated errors	13
Table 2: Percent errors of predicted x_D	15
Table 3: Reflux Ratio Errors	23
Table 4: LINEST data of fitted polynomial	24
Table 5: GC data of concentrations in reboiler over time	28
Table 6: GC data of concentrations in distillate over time	28
Table 7: Online VLE data for acetone/1-butanol system [4]	29

Batch Distillation

Objectives

The purpose of this lab is to understand Batch distillation of a binary system, Acetone and Butanol. We assume that our system has established total reflux at the beginning of the experiment. Based off boiling temperatures, Acetone is more volatile than Butanol. Hence, we expect our product to be mostly Acetone. We will analyze the process by using the Rayleigh equation and apply a McCabe Thiele diagram analysis. We assume that the column policy is constant reflux and that relative volatility is constant throughout the experiment duration.

Introduction

Distillation is a widely used method for separating chemicals. Batch distillation is a version of distillation where the column is pre-charged with a feed and is run on a batch basis. It is widely used in pharmaceutical, food, and biochemical industries [1]. It is run at an unsteady state since the system is not continuously fed and is limited with its initial charge. It is useful when the product is of high value with small batch operations. Its components are versatile and may be multipurposed.

In this experiment, we will explore batch distillation by separating Acetone and butanol. Our more volatile product, Acetone, is expected to be purified to a certain extent with percent butanol impurity. We will sample the distillate and reboiler compositions using Gas Chromatography (GC). We will observe how composition changed as well as the holdup in the reboiler and perform a McCabe Thiele analysis to help characterize the data and deduce a scaling of the apparatus.

Theory

The following are the assumptions made throughout our theoretical analysis.

Assumptions:

1. Constant Molecular weight and Density during the elapsed time.
2. Constant molar overflow which assumes constant liquid and vapor molar flow rates along the column.
3. Zero Holdup in the column.
4. Total Reflux achieved initially before operation.
5. Constant reflux throughout operation.
6. Constant Volatility.

Development of Rayleigh Equation

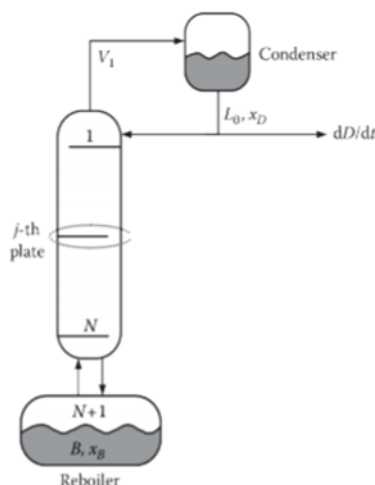


Figure 1: Simple Batch Distillation Apparatus [1]

Batch distillation consists of a reboiler at the bottom, condenser on top connected by a column as seen in Figure (1). The reboiler heats up the mixture, binary in our case, which leads to vapors rising through the column and condensing at the condenser. The column may be a single stage, multistage, or packed. Our experiment consists of a reboiler, four-stage column, and a total condenser. To understand the relationship between how the moles, change in the reboiler and its composition, we develop a relationship through a mass balance of a single component in the reboiler at a given time [1]. The resulting equation is called the Rayleigh equation.

The mass balance of Acetone in the reboiler.

$$n_A = x_B * n \quad (1)$$

Where

x_B = mole fraction of Acetone in the binary mixture.

n = Total moles in the reboiler [mol].

Since the composition is changing and the number of moles in the reboiler are changing, equation (2) is given.

$$dn_A = d(x_B * n) \quad (2)$$

Where

dn_A = The moles lost by the reboiler due to distillate [mol].

Therefore, the number of moles of Acetone lost in the distillate is equal to the change of the number of moles of Acetone in the Reboiler.

$$y * dn = dn_A = d(x_B * n) = dx_B * n + x_B * dn \quad (3)$$

Where

y = composition of Acetone in the condensate.

dn = loss of moles from the condensate to the distillate [mol].

By separating variables in equation (3) by having compositions on one side and number of moles on the other we result in equation (4).

$$\frac{dn}{n} = \frac{dx_B}{y-x_B} \quad (4)$$

Since the compositions and moles are time dependent, we integrate equation (4) from $t=0$ to $t=t$.

$$\int_{n(0)}^{n(t)} \frac{dn}{n} = \int_{x(0)}^{x(t)} \frac{dx_B}{y-x_B} \quad (5)$$

Solving the integral results in equation (6).

$$\ln\left(\frac{nt}{n_0}\right) = \int_{x(0)}^{x(t)} \frac{dx_B}{y-x_B} \quad (6)$$

We assume that the molecular weight and density are constant from $t = 0$ to t .

$$n = \frac{m}{MW} \quad (7)$$

Where

n = number of moles [mol]

m = mass [Kg]

MW = molecular Weight

Therefore,

$$\left(\frac{n * MW}{\rho}\right) = \left(\frac{m}{\rho}\right) = H \quad (8)$$

Where

ρ = Density [Kg/L]

H = Holdup Volume in the reboiler [L]

Finally, the Rayleigh equation (6) is expressed in terms of holdup.

$$\ln\left(\frac{Ht}{H_0}\right) = \int_{x(0)}^{x(t)} \frac{dx_B}{x_D-x_B} \quad (9)$$

Since the distillate composition is the same as the condensate composition and we have a full condenser, we can substitute y by the Acetone mole fraction in the distillate, x_D .

$$\ln\left(\frac{Ht}{H_0}\right) = \int_{x(0)}^{x(t)} \frac{dx_B}{x_D-x_B} \quad (10)$$

Constant Reflux – Average Distillate Composition and Batch Time

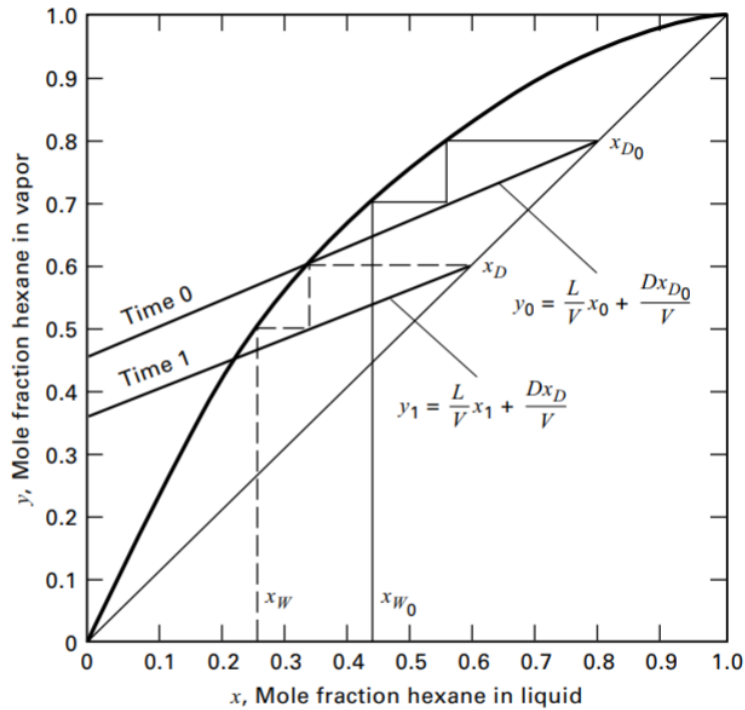


Figure 2: Constant Reflux at multiple times [2]

Based off the work of Smoker and Rose, equations (11) and (12) represent the average distillate composition in terms of holdup and batch time for a constant reflux operation [2]. They utilized the McCabe Thiele diagram of the light key (LK) for their analysis. By stepping off stages and constructing operating lines that are parallel, where each line represented a time, due to a constant slope (Constant reflux) [2]. They were able to develop a time dependent series of x_D and x_B as seen in figure (2).

$$x_{d,avg} = \frac{x_{bi} - H_f * x_{bf}}{H_i - H_f} \quad (11)$$

$$\vartheta = \frac{H_i - H_f}{V \left(1 - \frac{L}{V}\right)} = \frac{R+1}{V} * (H_i - H_f) \quad (12)$$

Where

ϑ = Batch time [s]

R = Reflux ratio

V = Boilup rate [m^3/s]

L = Liquid rate [m^3/s]

Constant Distillate Composition – Batch Time and Final Batch Quantity

Based off the work of Bogart on constant distillate operation, a batch time and final batch quantity can be calculated [2]. Bogart assumes constant molar overflow and no liquid holdup. By rearranging equation (11), we express holdup in the reboiler in terms of initial reboiler holdup and mole fraction of the distillate and reboiler.

$$H = H_i * \left(\frac{x_D - x_{Bi}}{x_D - x_{Bf}} \right) \quad (13)$$

Where

x_D = Constant distillate composition.

x_{Bi} = Initial reboiler composition.

x_{Bf} = Reboiler composition at holdup H.

Differentiating both sides yields equation (14).

$$\frac{dH}{dt} = H_i * \left(\frac{x_D - x_{Bi}}{x_D - x_{Bf}} \right) \left(\frac{dx_b}{dt} \right) \quad (14)$$

Assuming constant overflow, distillation rate is the rate of loss from the reboiler, equation (15).

$$\frac{-dH}{dt} = (V - L) = \frac{dD}{dt} \quad (15)$$

Where

D = distillate amount (not rate) [m^3]

Substituting equation (15) in to equation (14).

$$t = H_i * \frac{x_D - x_{Bi}}{V} * \int_{x_{Bf}}^{x_{Bi}} \frac{dx_b}{\left(1 - \frac{L}{V}\right) * (x_D - x_B)^2} \quad (16)$$

Finally, the batch quantity is given by equation (17)

$$H_i - H_f = H_i * \frac{x_{Bi} - x_{Bf}}{x_D - x_{Bf}} \quad (17)$$

McCabe Thiele Graphical Analysis

The McCabe Thiele method is a powerful graphical method in understanding binary distillation systems. The graph used in the analysis consists of the y-axis being the mole fraction of the more volatile species in the Vapor phase while the x-axis is the mole fraction of the more volatile species in the liquid phase. In our case, the species of question is Acetone. On the graph, we have a 45° line where the mole fractions are equal, an operating line and an equilibrium curve [2]. For subscripts, they indicate the source stage where the column is numbered from top to bottom in ascending order (i.e 1,2,..etc)

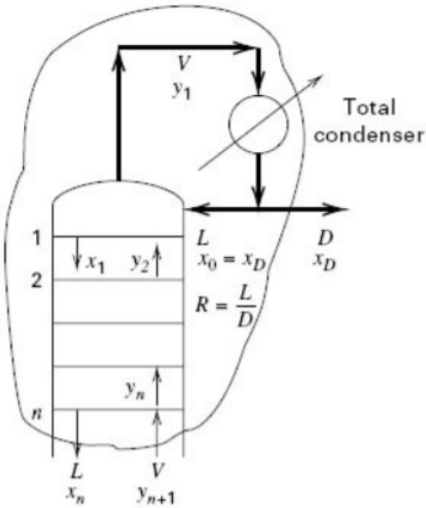


Figure 3: Rectifying Section Mass Balance Analysis [2]

To find the operating line that represents our process, we perform a mass balance across the rectifying section as shown in figure (3). Where we have vapors entering from the reboiler, liquid entering the reboiler and distillate leaving from the top of the column.

$$in = out \quad (18)$$

$$V = L + D \quad (19)$$

Where

V = Vapor molar Flow rate [mol/sec]

L = liquid molar flow rate [mol/sec]

D = Distillate molar flow rate [mol/sec]

By applying a material balance with respect to the light key on the same system defined in figure (3), we get equation (20).

$$V_{n+1} * y_{n+1} = L_n * x_n + D * x_D \quad (20)$$

Rearranging for y_{n+1} . we get the rectifying section operating line, equation (21).

$$y_{n+1} = (L_n/V_{n+1}) * x_n + (D/V_{n+1}) * x_D \quad (21)$$

Since we assume constant molar overflow, we re-write equation (21).

$$y = (L/V) * x + (D/V) * x_D \quad (22)$$

Then, we define a reflux ratio, R, to be the ratio of Liquid flow rate to the distillate flow rate.

$$R = \frac{L}{D} \quad (23)$$

$$\frac{L}{V} = \frac{L}{L+D} = \frac{\frac{L}{D}}{\frac{L}{D}+1} = \frac{R}{R+1} \quad (24)$$

$$\frac{D}{V} = \frac{D}{L+D} = \left(\frac{1}{R+1} \right) \quad (25)$$

Therefore, the operating line in terms of reflux ratio, R is illustrated in equation (26).

$$y = \left(\frac{R}{R+1} \right) * x + \left(\frac{1}{R+1} \right) * x_D \quad (26)$$

A summary of the rectifying section McCabe Thiele diagram can be seen in figure (4).

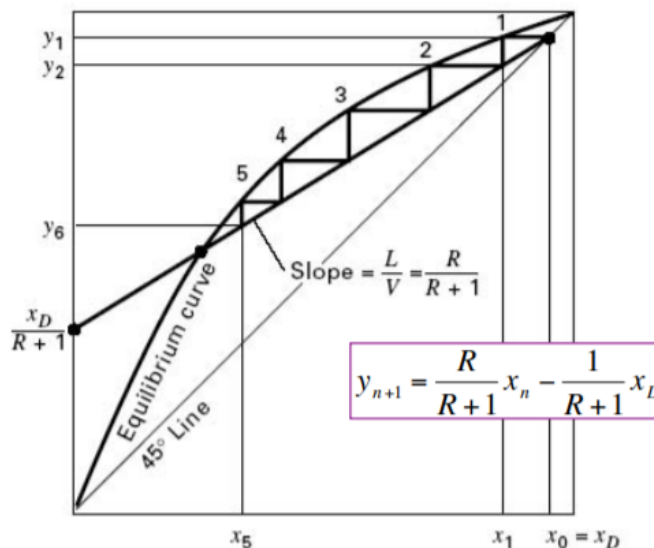


Figure 4: Rectifying Section McCabe Thiele Diagram [2]

Relative Volatility

Relative volatility is the ratio of the K-value of the light key to the heavy key.

$$\alpha = K_{LK}/K_{HK} \quad (27)$$

Where in our case

LK = Acetone

HK = Butanol

Assuming the mixture is an ideal solution, it obeys Raoult's law [2].

$$K_i = y_i/x_i \quad (28)$$

Where

Y_i = Vapor composition of species i

X_i = liquid composition species i

Therefore, relative volatility can be expressed in terms of the ratio of vapor pressure of the two components.

$$\alpha = \frac{\frac{y_{LK}}{x_{LK}}}{\frac{y_{HK}}{x_{HK}}} = \frac{y_{LK} * (1 - x_{LK})}{x_{LK} * (1 - y_{LK})} \quad (29)$$

Where

LK = Acetone

Solving for y_1 yields equation (30)

$$y_{LK} = \frac{\alpha * x_{LK}}{1 + x_{LK} * (\alpha - 1)} \quad (30)$$

Relative volatility is an indicator of how well a separation can be done. The higher the volatility, the more of the volatile component is in the vapor compared to the heavy key. Changing relative volatility and its effect on equilibrium is seen in figure (5).

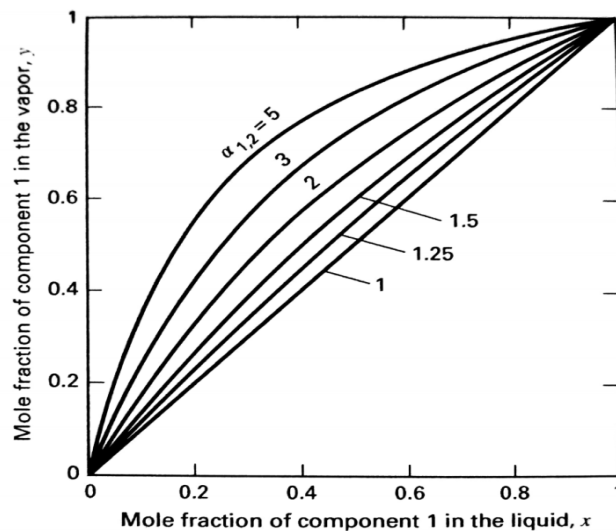


Figure 5: Relative Volatility Effect on Equilibrium Curve [2]

Integration of Rayleigh Equation and Incorporation of Relative Volatility

Integration of the Rayleigh equation (10) for a binary mixture with constant relative volatility results in equation (31).

$$\ln \left(\frac{H_0}{H} \right) = \frac{1}{\alpha - 1} * \left[\ln \left(\frac{x_0}{x} \right) + \alpha * \ln \left(\frac{1 - x}{1 - x_0} \right) \right] \quad (31)$$

We can also derive an equation that deems useful in understanding the effect of relative volatility in a batch distillation, assuming constant relative volatility.

Applying a mass balance,

$$\frac{dH_{LK}}{dt} = \frac{d}{dt} * (H * x_{B,LK}) = -D * y_{D,LK} \quad (32)$$

Where

H = Holdup in the reboiler [m^3]

D = Distillate volume [m^3]

LK = Acetone

We can also apply the same mass balance for the heavy key.

Then, we can present holdup ratio in terms of vapor composition ratio.

$$\frac{dH_{LK}}{dH_{HK}} = \frac{y_{D,LK}}{y_{D,HK}} \quad (33)$$

combining equation (29) and (33), then integration from initial reboiler charge condition yields equation (34).

$$\ln \left(\frac{H_{A,f}}{H_{A,i}} \right) = \alpha * \ln \left(\frac{H_{B,f}}{H_{B,i}} \right) \quad (31)$$

Where

A = Acetone

B = Butanol

i = initial

f = final

α = Relative Volatility

Boil up Calculation

Performing a mass balance, we arrive to equation (32). It relates the change of the holdup with respect to time [2].

$$\frac{dH}{dt} = L - V \quad (31)$$

$$dH = (L - V)dt \quad (32)$$

Integrating equation (32) from Initial to final condition.

$$H_f = H_i + (L - V) * t = H_i - \frac{V}{R+1} * t \quad (33)$$

Where

H_f = Final Holdup [L]

H_i = Initial Holdup [L]

L = Liquid Flowrate [L/min]

V = Boil up [L/min]

t = Time [min]

R = Reflux Ratio

Results/Data

Table 1: Key batch distillation values and associated errors

		Value	Error
Relative Volatility	Experimental	2.42	1.13
	Online Data	2.32	n/a
Reflux Ratio		15.875	0.254
x_d , predicted (mole fraction of acetone)	t=5	0.867	0.075
	t=10	0.840	0.075
	t=20	0.793	0.075
	t=30	0.708	0.075
	t=40	0.642	0.075
	t=50	0.554	0.075
Rayleigh Equation	LHS	-0.251	0.034
	RHS	-0.198	0.513

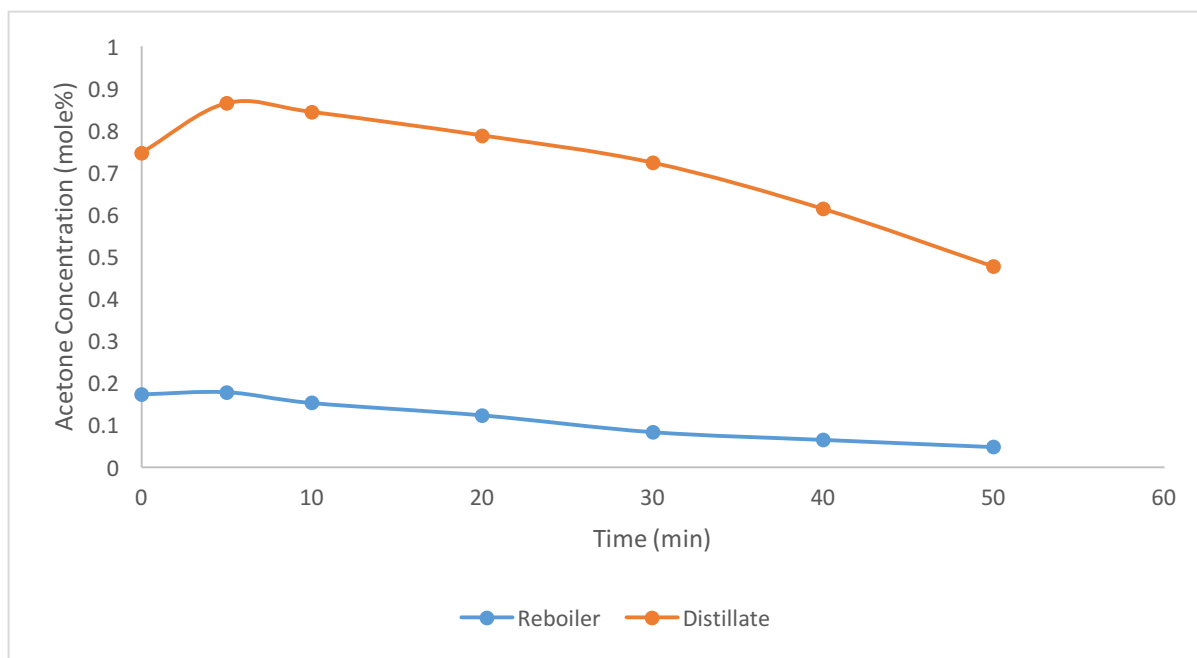


Figure 6: Acetone concentration over time

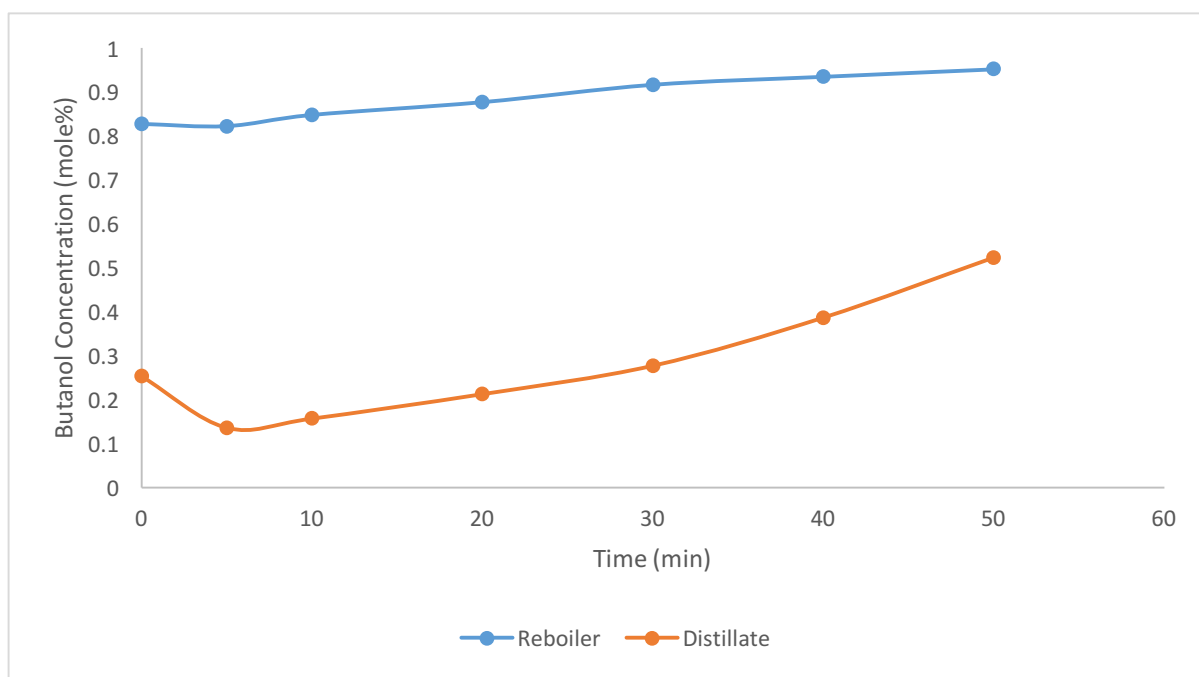


Figure 7: n-Butanol concentration over time

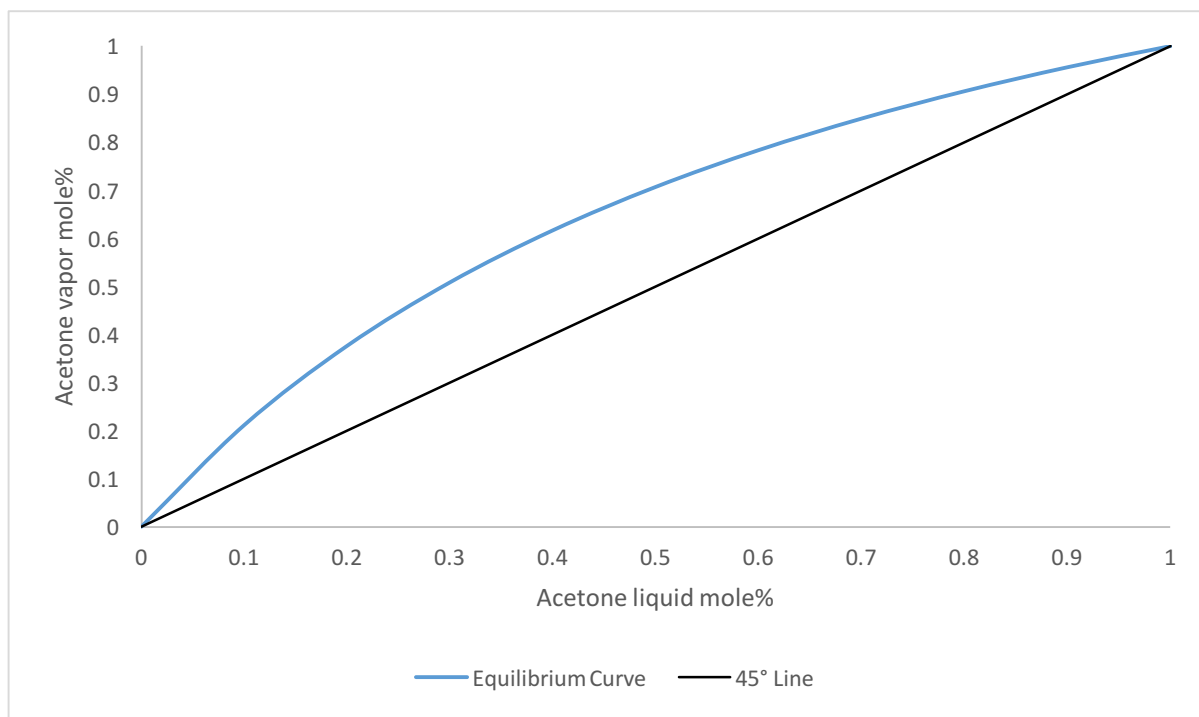


Figure 8: x-y graph of experimental acetone/n-butanol system

Table 2: Percent errors of predicted x_D

Time (min)	0	5	10	20	30	40	50
%error of $x_{D,predicted}$	0	0.398	0.381	0.738	2.020	4.626	16.272

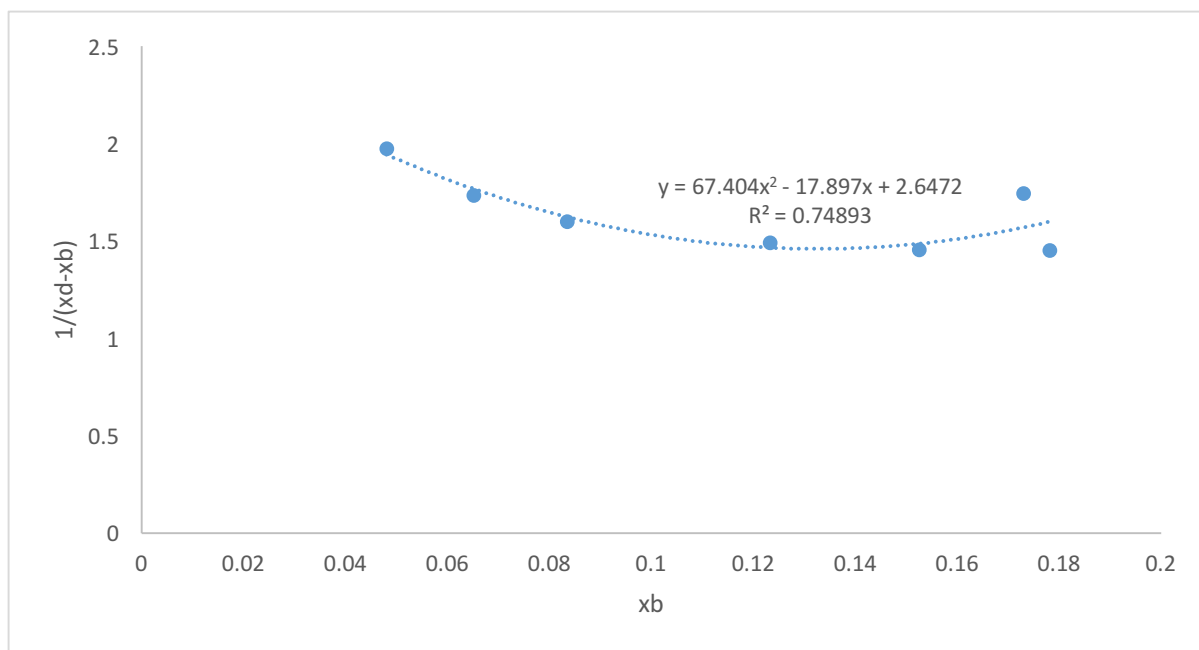


Figure 9: $1/(x_D - x_B)$ vs x_B graph for integrating RHS of Rayleigh equation

Analysis

As seen in Figure 6, an immediate discrepancy is noticed. The acetone concentration in the distillate at total reflux is lower than the 5-minute distillation concentration. This shows that total reflux was not reached since the concentration of acetone over time in the distillate should have a downward trend as it is the more volatile substance. Another reason for the acetone concentration to be higher during total reflux is that the boiling point of acetone is lower, so more acetone should be in the distillate.

The next step was to calculate the relative volatility. For this, the assumption that the total reflux data was good was made, not the best assumption, but the assumption allowed for results to be obtained. Since total reflux is assumed, the reflux ratio, R , was not required in the

calculations (see Sample Calculations). Therefore, allowing for the relative volatility to be solved for based on the data, then fit to minimize the residual error, obtaining a relative volatility of 2.42 ± 1.13 . This value, along with its error range, overlap with the literature value, 2.32 [3]. Since, only one trial was used to get the relative volatility the residual could potentially be minimized to 0. If more trials were done, the average value and residual error could be different.

The reflux ratio, R , was then calculated using the concentrations of the distillate and reboiler along with the found relative volatility. A similar calculation method was used as for the relative volatility, but the operating line had to be accounted for when moving between stages due to the reactor not being at total reflux. Another assumption used is that the reflux ratio did not change throughout the experiment, which should be valid since the valve location was not changed as the distillation occurred. Using the different times and concentrations, an initial R of 1 was assumed then run through each time to get a y_1 for each time. The squared residual error was then calculated and minimized to find the R that fit the data the best. The R for the results was 15.875 ± 0.253 . The predicted concentrations for each time and their associated errors can be found in Table 1, along with the R and relative volatility values.

The next part is to calculate the Rayleigh equation to ensure that the LHS and the RHS of the equation are equal to one another. Assumptions of the Rayleigh equation can be seen in the Theory. The results of this experiment show that the LHS of the Rayleigh equation is -0.251 ± 0.034 and the RHS of the Rayleigh equation is -0.198 ± 0.513 . Although the RHS of the equation has a range that overlaps the LHS of the equation, the probability of the RHS of the equation to be the same as the LHS of the equation is pretty low due to the huge imprecision of the RHS value. Hence, for this experiment, it can be assumed that the Rayleigh equation was unable to close.

Reasons that the Rayleigh equation was unable to close include potentially invalid assumptions including constant densities. Since the operating temperatures of the distillation column varies quite a lot ($\sim 40^\circ\text{C}$ in the distillate), it is highly likely that the densities of both the acetone and the butanol have changed, especially since both of the substances are relatively small and volatile. Another reason for the Rayleigh equation not closing is because of the constant removal of samples from both the distillate and the reboiler as GC samples. Although there was an attempt to reduce this discrepancy by factoring in the removed volume into the final reboiler volume, the Rayleigh equation was still unable to close. This was probably because the volume that was removed was no longer subject to distillation, hence the volume removed is disproportionate to the volume that would have been if no holdup was removed. Lastly, because total reflux is not reached, the error associated with using a non-total reflux data and basing the experiment off of it will compound the error such that the RHS of the equation is inaccurate.

Conclusions

The aim of this experiment is to use the Rayleigh equation to characterize a batch distillation to the distillation of acetone and n-butanol and then determine if scaling up is possible based on values obtained. From this experiment, it is apparent that the Rayleigh equation is unable to close, which suggests errors, which can be seen in the Error Analysis section. Perhaps the largest source of error was that total reflux was not reached, hence all subsequent calculations that assumed total reflux was reached were technically invalid, despite the experimental relative volatility agreeing with literature value. In other words, there is an inherent error in all values related to total reflux.

The design question was deemed undoable due to a lack of information as well as unrealistic specifications to accomplish the task (see Design Question Details). A scale-up could be possible if the experiment was redone such that the total reflux is reached. Moreover, redoing the experiment with the same initial concentrations as described in the design question will help relate the experiment to the scale-up better.

References

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Appendices

Sample Calculations

To find *n*-butanol concentrations:

$$c_{\text{butanol}} = 1 - c_{\text{acetone}}$$

$$0.8269 = 1 - 0.1731$$

To find relative volatility, α , use data at total reflux (also applied to data found in literature):

$$x_3 = x_B = 0.1731$$

$$y_3 = \frac{\alpha x_3}{1 + (\alpha - 1)x_3}$$

$$x_2 = y_3$$

$$y_2 = \frac{\alpha x_2}{1 + (\alpha - 1)x_2}$$

$$x_1 = y_2$$

$$y_1 = \frac{\alpha x_1}{1 + (\alpha - 1)x_1}$$

$$0.7467 = x_D = y_1$$

Using Excel's Solver tool to reduce the difference between x_D and y_1 to 0, which works similar to combining and rearranging the above equations such that $\alpha = f(x_B, x_D)$:

$$\alpha = 2.418$$

The relative volatility can then be used to construct an equilibrium curve with the following general equation, where $0 \leq x \leq 1$:

$$y = \frac{\alpha x}{1 + (\alpha - 1)x}$$

To estimate the reflux ratio, R , use data after total reflux (using data at $t = 5$ min):

$$x_D = 0.8640$$

$$x_3 = x_B = 0.1782$$

$$y_3 = \frac{\alpha x_3}{1 + (\alpha - 1)x_3}$$

$$x_2 = \frac{R + 1}{R} y_3 + \frac{x_D}{R}$$

$$y_2 = \frac{\alpha x_2}{1 + (\alpha - 1)x_2}$$

$$x_1 = \frac{R + 1}{R} y_2 + \frac{x_D}{R}$$

$$y_1 = \frac{\alpha x_1}{1 + (\alpha - 1)x_1}$$

$$x_{D,predicted} = y_1$$

Using Excel's Solver tool to minimize the total residual (see Error Analysis) between x_D and $x_{D,predicted}$ by altering the reflux ratio, R :

$$R = 15.875$$

To calculate Rayleigh's equation:

$$\ln\left(\frac{H_f}{H_i}\right) = \int_{x_B(0)}^{x_B(t)} \frac{dx_B}{x_D - x_B}$$

RHS:

The volume removed from the system for GC analysis is added back into the final holdup volume such that:

$$H_f = H_{f,measured} + H_{removed}$$

$$3.6575 \text{ L} = 3.500 \text{ L} + 0.1575 \text{ L}$$

$$RHS = \ln\left(\frac{3.6575 \text{ L}}{4.700 \text{ L}}\right) = -0.251$$

LHS:

By graphing x_B against $1/(x_D - x_B)$, a polynomial curve, see figure (9), was fitted to the data, which can be used to calculate the area under the curve (=LHS):

$$LHS = \int_{0.1731}^{0.0481} (67.404x_B^2 - 17.897x_B + 2.647)dx_B = -0.198$$

Error Sample Calculations

Preface:

General error propagation equation:

$$g = F(x, y, z \dots)$$

$$\varepsilon_g = \sqrt{\left(\frac{\partial F}{\partial x}\right)^2 \varepsilon_x^2 + \left(\frac{\partial F}{\partial y}\right)^2 \varepsilon_y^2 + \left(\frac{\partial F}{\partial z}\right)^2 \varepsilon_z^2 + \dots}$$

Due to GC pipet handling issues as well as inconsistencies, the errors for all measured concentrations obtained from the GC are 0.2 (see Error Analysis).

To find the error for relative volatility, α , using MATLAB:

Due to GC pipet handling issues as well as inconsistencies, the errors for all measured concentrations obtained from the GC are 0.2 (see Error Analysis).

% isolate alpha from combined equations (see Sample Calculations)

`clear`

`syms y x a`

`eqn = (a^3 * x)/(1 - x + a^3 * x) == y;`

`v_a = solve(eqn,a)`

% using the real solution from v_a as f and creating a function that accepts x and y (concentrations of acetone in the reboiler and distillate at total reflux respectively) to calculate partial derivatives

`clear`

`syms x y`

`f = ((y - x*y)/(x - x*y))^(1/3);`

`dfdy = diff(f,y);`

`dfdx = diff(f,x);`

`df = [dfdy dfdx]';`

`DF = matlabFunction(df, 'file', 'alpha');`

% use the generated function, alpha, to obtain partial derivatives to


```
(a*x/(1+x*(a-1))) - x/R)) - x/R)) == y;

v_R = solve(eqn,R)

% use the solution from v_R
clear
syms x y a

f = v_R

dfdy = diff(f,y);
dfdx = diff(f,x);
dfda = diff(f,a);

df = [dfdy dfdx dfda]';
DF = matlabFunction(df,'file','refluxratio');

% set variables then use the function generated to obtain partial
derivatives to find error using general error propagation equation
% x = measured distillate concentrations; y = predicted distillate
concentrations; ey = error of y;
x = [0.1782
      0.1526
      0.1233
      0.0835
      0.0652
      0.0481];

y = [0.86743804
      0.840089837
      0.793411065
      0.708392101
      0.641882973
      0.554383186];

ey = 0.075334074;

error = zeros(length(x),1);

for i = 1:length(x)
    df = refluxratio(2.414843341,x(i),y(i));

    dfdy = df(1);
```

```
dfdx = df(2);
dfda = df(3);

error(i) = sqrt(dfdy^2 * ey(i)^2 + dfdx^2 * 0.2^2 + dfda^2 *
1.1247^2);
end
```

The table below contains the reflux ratio errors for each trial, which are then averaged (the average is used as the error for the reflux ratio):

Table 3: Reflux Ratio Errors

Time (min)	5	10	20	30	40	50
Error for R	0.147	0.162	0.189	0.259	0.326	0.441
Average	0.2539					

To calculate error for Rayleigh's equation:

LHS:

```
% define Variables
syms Hi Hfm Hfr

% function form, f
f = log((Hfm+Hfr)/Hi);

% Find Partial of function, f
dfdHi = diff(f,Hi);
dfdHfr = diff(f,Hfr);
dfdHfm = diff(f,Hfm);

df=[ dfdHi , dfdHfr , dfdHfm ]';
Df = matlabFunction(df,'file','DFM');

de = DFM(3.5,0.1575,4.7);

E_Hi = 0.1; %[L]
E_Hfm = 0.1; %[L]
E_Hfr = 0.2/1000; %[L]
```

```
Error = sqrt( (de(1)^2)*(E_Hi)^2 + E_Hfr^2*de(2)^2 + (E_Hfm^2)*(de(3))^2 );
```

$$LHS = f = \ln\left(\frac{H_{fm} + H_{fr}}{H_i}\right)$$

Where

H_{fm} = final measured Holdup [L]

H_{fr} = Removed Sampling Holdup [L]

H_i = Initial holdup [L]

Error in the LHS of the Rayleigh equation = 0.0346

RHS:

The fitted polynomial has the following form:

$$y = ax^2 + bx + c$$

The values obtained from LINEST are tabulated below:

Table 4: LINEST data of fitted polynomial

	a	b	c
Magnitude	67.40411853	-17.89655162	2.647210025
Error	29.54920606	6.862536419	0.341129221

MATLAB is used to calculate the error for the RHS of the Rayleigh equation:

```
% create function to calculate partial derivatives where a, b, c are the
polynomial constants. The function, f, is integrating the polynomial in terms
of xb, the acetone concentration in the reboiler, from xbi, xb initial, to
xbf, xb final.
```

```
clear
```

```
syms a b c xb xbi xbf
```

```
f = int(a*xb^2 + b*xb + c, xbi, xbf);
```

```
dfda = diff(f,a);
```

```
dfdb = diff(f,b);
```

```
dfdc = diff(f,c);
```

```
dfdxbi = diff(f,xbi);
```

```
dfdxbf = diff(f,xbf);
```

```
df = [dfda dfdb dfdc dfdxbi dfdxbf]';
```

```
DF = matlabFunction(df,'file','distillationint');
```

```
% calculate the partial derivatives to find error using the general error
propagation equation
```

```
df = distillationint(67.40411853,-17.89655162,2.647210025,0.0481,0.1731);
```

```
dfda = df(1);
```

```
dfdb = df(2);
```

```
dfdc = df(3);
```

```
dfdxbi = df(4);
```

```
dfdxbf = df(5);
```

$$\text{error} = \sqrt{(\text{dfda}^2 * 29.54920606^2 + \text{dfdb}^2 * 6.862536419^2 + \text{dfdc}^2 * 0.341129221^2 + \text{dfdxbi}^2 * 0.2^2 + \text{dfdxbf}^2 * 0.2^2)}$$

$$\varepsilon_{RHS} = 0.5152$$

Error Analysis

The GC concentration error was assumed to be 0.2. The error could have been up to 50% or as low as 5% to 10% due to just injection errors [4]. We felt that the best estimate would be around 20% or 0.2 as an initial estimate. This is most likely an overestimate since the GC can produce results up to the ten-thousandth and the lowest error would be 0.05.

The error for the relative volatility along with subsequent related values were calculated assuming that total reflux was reached at time = 0. For this reason, the error associated with an incorrect total reflux was not factored into error calculations. The simple reason is that the data for true total reflux concentrations is unknown. This suggests that although error values for certain values may be low, they only represent errors for this experiment.

Time is also a source of error. Since the experiment entails two different people collecting samples at the same specific times, errors may occur. Moreover, sample collecting occurs over the span of several seconds. While it is quick for the reboiler, the time it takes to collect samples for the distillate is relatively long. The errors for these time-based measurements can be accounted for in the GC error since the GC errors were deliberately overestimated.

The percent error for the predicted x_D 's were calculated individually based off their measured counterparts. The 95% confidence absolute error were calculated collectively based off the sum squared error (see Error Analysis Calculations), hence the reason why all of the predicted x_D 's have the same absolute error. The absolute error for the predicted concentrations are therefore not indicative of an individual estimates error, but rather the error for the collective estimates as well as the method for calculating the reflux ratio.

A big source of error occurs when calculating the Rayleigh equation. Figure (9) shows fitted data to calculate the RHS of the Rayleigh equation with an R^2 value of 0.74893. Since the data was not ideally fitted, the errors associated with the LINEST values, see table (4), are relatively big. The reason for a poor fit was due to the total reflux data point, which was not according to the apparent trend (see Data Analysis). As a result, the error for the RHS of the Rayleigh equation is very large, see table (1).

For future experiments, it is best to wait longer to ensure that total reflux is achieved before continuing with the rest of the experiment. Moreover, attempting to minimize the volume removed from the distillation column for GC analysis may also help ensure that the Rayleigh equation will close.

Design Questions Details

For the scale-up based on the experimental data, we do not think it is possible to reach 90% acetone recovery reasonably. First, we assumed the relative volatility was the same as for the experiment, 2.415. We then attempted to change the Reflux Ratio (R) and the number of theoretical stages (n). We chose to fix the number of stages to 4, and after continuously adjusting the reflux ratio and number of theoretical stages, the reflux ratio was determined to be 6.64. For a realistic scaled-up column, the number stages we determined was far too low as that would mean that its diameter would be massive and the height of each tray could also be massive. Either way the column would be unrealistic. Moreover, the vapor rate, V, depends on the tray diameter as well as the amount of heating applied at the reboiler. If tray diameter is too large, uneven heating as well as separation would be lower since vapor rate would be very low. Lastly, the amount of energy required to maintain the heat for each batch would be unrealistic as well due to cost constraints.

To scale-up the column more realistically, the same initial compositions of the feed would most likely be required. We would need to reach total reflux during the experiment to guarantee an accurate relative volatility. Additional information of the scaled-up column would also be required such as its diameter, some recommended specifications (e.g. vapor flow rate and batch time).

Script:

%Our Data

% t = [0 5 10 20 30 40 50];

% xb = [.1731 .1782 .1562 .1233 .0835 .0652 .0481];

% xd = [.7467 .8640 .8433 .7876 .7230 .6135 .4768];

%Given

Vi = 10000;

xb0 = .3;

xd = 0.9;

%Assume

n = 4;

R = 6.64;
alpha = 2.415;

%Equations

```
comp_mat = zeros(n,2);      %matrix of composition data
comp_mat(1,1) = xb0;
for i = 1:n-1
    comp_mat(i,2) = EC(comp_mat(i,1),alpha);
    comp_mat(i+1,1) = OL(comp_mat(i,2),xd,R);
end
comp_mat(n,2) = EC(comp_mat(n,1),alpha)
```

```
function y = EC(x,alpha)
y = alpha * x / (1+ (alpha-1) * x);
end
```

```
function x = OL(y,xd,R)
x = (R+1)/R * y - xd / R;
end
```

>> Distillation_Design_Question

comp_mat =

```
0.3000  0.5086
0.4695  0.6812
0.6593  0.8238
0.8161  0.9147
```

Raw Data

Table 5: GC data of concentrations in reboiler over time

Time (min)	acetone, x	butanol, x'	Temperature (°C)
Initial	0.1955	0.8045	20.9
0 (Total Reflux)	0.1731	0.8269	82
5	0.1782	0.8218	84
10	0.1526	0.8474	88
20	0.1233	0.8767	91
30	0.0835	0.9165	96
40	0.0652	0.9348	100
50	0.0481	0.9519	104

Table 6: GC data of concentrations in distillate over time

Time (min)	Distillate		
	acetone, y	butanol, y'	Temperature (°C)
0 (Total Reflux)	0.7467	0.2533	59
5	0.8640	0.136	61
10	0.8433	0.1567	64
20	0.7876	0.2124	66
30	0.7230	0.277	71
40	0.6135	0.3865	82
50	0.4768	0.5232	95
Final (51:25)	0.7388	0.2612	

Table 7: Online VLE data for acetone/1-butanol system [4]

°C	xi	yi
115.15	0.01	0.105
112.15	0.025	0.225
107.25	0.05	0.405
103.95	0.07	0.472
91.85	0.154	0.698
87.05	0.198	0.75
85.55	0.215	0.773
81.85	0.265	0.809
78.95	0.312	0.837
68.75	0.535	0.93
66.35	0.607	0.943
64.65	0.659	0.951
61.75	0.763	0.972
60.15	0.819	0.978
58.25	0.897	0.988

* blue highlights indicate experimental operating temperatures of reboiler and distillate at total reflux ($T_B = 82\text{ }^{\circ}\text{C}$, $T_D = 59\text{ }^{\circ}\text{C}$)

Experimental Procedure

No deviations from the lab manual [5].

Laboratory Safety Analysis

Basic Steps	Potential Hazards	Controls

I understand & will adhere to the hazards & controls as described in this LSA including the unusual and area hazards. I will contact the TA or Lab Manager prior to continuing work, if conditions change or new hazards are introduced.

I understand I have the authority and responsibility to stop work I believe to be unsafe.

Student Name (please print) Muhammad Samir Signature [Signature] Date 03-21-2019

for Jonathan Lin [Signature]

Muhammad Elris [Signature]

Teaching Assistant Yimin Wang Signature [Signature]

USC Viterbi <i>The Mork Family Department of Chemical Engineering and Materials Science</i>		Laboratory Safety Analysis (LSA)		Date : 3/21/17
EXPERIMENT/ACTIVITY NAME: Batch Distillation		BLDG/AREA LOCATION(s): PCE 104/105		Undergraduate Chemical Engineering Laboratory
DEPARTMENT/GROUP NAME CHE 444 Lab:		EMERGENCY INFORMATION: USC EHS/DPS 213-740-4321 Provide Location and nature of the emergency (injury, spill, fire, etc.		Contact Information: Lab Manager: Tina Silva (c) 818-645-4187 Instructor: Robert Young (c) 714-504-4334
REQUIRED PERSONAL PROTECTIVE EQUIPMENT FOR EXPERIMENT				
<input checked="" type="checkbox"/> safety glasses <input checked="" type="checkbox"/> lab coat <input type="checkbox"/> nitrile/latex gloves <input type="checkbox"/> other _____ <input type="checkbox"/> face shield <input type="checkbox"/> chemical apron <input checked="" type="checkbox"/> thermal gloves <input type="checkbox"/> other _____ <input type="checkbox"/> hearing protection <input type="checkbox"/> safety shoes <input type="checkbox"/> leather gloves <input type="checkbox"/> other _____				
Basic Steps	Potential Hazards	Controls		
Moving valves	Acetone and n-Butanol are flammable	Opening or closing the wrong valves or the right valve at the wrong time can result in loss containment and exposure of hazardous. Confirm with procedure actions to be taken BEFORE they are implemented.		
Collecting samples from reboiler and distillate product stream.	Acetone and Butanol are mild skin irritants	Wearing nitrile gloves and safety goggles at all times. Long pants as well as toe covered shoes.		
Collecting samples from reboiler and distillate product stream.	Sample between 50C and 120C – Contact without adequate protection can result in thermal skin irritation or burns	Working under the fumehood when preparing samples.		
Use of syringe/sharps to transport the sample Running GC	Puncture injury from needle	Wearing the heat resistant gloves when collecting samples		
		While wearing nitrile gloves protects from chemical exposure, they will not prevent puncture injuries. Keeps hands/body parts out of the "Line of Fire" of the needle. When not in use, point the needle away from where people could be punctured.		

Group Distribution of Effort

Executive Summary- Mohammed Idriss

List of Figures & Tables - Mohammed Idriss

Objectives - Mohammed Idriss

Introduction - Mohammed Idriss

Theory – Mohammed Idriss

Results/Data – Chih-Hsien Lin

Analysis – Chih-Hsien Lin & Theodore Somekh

Conclusion – Chih-Hsien Lin & Theodore Somekh

Sample Calculations - Chih-Hsien Lin & Theodore Somekh

Error Analysis - Chih-Hsien Lin & Theodore Somekh

Design Question – Theodore Somekh

Procedure – Theodore Somekh