University of San Carlos
Department of Chemical Engineering

CHE 4118L Chemical Engineering Laboratory Investigations 2

Experiment Plan

(Form CHE 4118L-1)

Prepared and submitted by:

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Experiment : **Distillation of Ethanol-Water in a Batch Distillation Column**

Objectives of the Experiment

1. Determine the average concentration of a specified volume of the distillate that is obtained from a feed mixture of known composition by operating the distillation unit at constant reflux.
2. Use experimental data to predict the time required to obtain a particular concentration of the distillate; and
3. Determine the total number of stages for the batch distillation process.

Methodological Framework

(For Objectives 1 through 3)

(Based on REAL-LIFE or ACTUAL conduct of experiment)

A. Calibration
   a. Preparation of Ethanol Concentration ranging from 0% (pure water) to 100% (pure ethanol) by weight by increments of 20% by weight concentration from analytical grade ethanol.
   b. Measurement of the refractive index of each prepared concentration.
   c. A plot of weight concentration (x) as a function of refractive index will be done along with the determination of the trendline of the system. Equation and correlation value (r^2) will be obtained.

B. Preparation
   a. Preparation of 27L with 30% w/w ethanol solution (technical grade).

C. Operation
   a. Pre-startup and start-up operations will be conducted
   b. Fully Opened Reflux Valve

i. Operation of distillation setup at total reflux for 20 minutes
ii. Time to fill upper product receiver from lower to upper mark and its volume will be recorded

c. One-sixth open Reflux Valve
   i. The time to fill upper product receiver at 1/6-open reflux and volume of distillate upon filling up product receiver from lower to upper mark was documented.
   ii. Ten milliliters of still and distillate sections will be obtained.
   iii. Samples of distillate and still will be collected every 5 minutes. Refractive Index per interval will be measured along with its temperature.
   iv. Operations halts when 3L of distillate is collected. **Actual time** $\left(t_{actual}\right)$ **of distillation** will be recorded.

d. Shutdown Operations will be performed

(Based on VIRTUAL experiment or PROVIDED DATA)

A. Extrapolation of Data
   a. Plots for time vs. weight concentration for distillate and bottoms and weight concentration vs. $\frac{1}{x_D - x_B}$ are given by the instructor beforehand.
   b. Using time vs. weight concentration plot provided, points are extrapolated for concentration at bottoms or still (x_B) and at distillate (x_D).
   c. The actual distillate time (t_{actual}) from the same plot will be extrapolated based on the last point of the time vs. weight concentration plot.
   d. The recorded time at total reflux and 1/6 opening reflux will be acquired.

Objective 1: Determine the average concentration of a specified volume of the distillate that is obtained from a feed mixture of known composition by operating the distillation unit at constant reflux.

A. Calculation of by weight concentration of still and distillate from calibration regression equation obtained.
B. Concentrations of Distillate and Still as a function of time were plotted
C. The plot of $\frac{1}{x_D - x_B}$ as function of x_B will be constructed and the polynomial equation trendline and correlation value r^2 is obtained.
D. Calculation of final amount of bottoms (B_f) will be done using Rayleigh Equation.
E. From total amount of Distillate $\left(D_{total}\right)$ and Bottoms at initial or feed $\left(B_0\right)$ and final $\left(B_f\right)$ and concentrations of bottoms at initial $\left(x_{B,0}\right)$ and final $\left(x_{B,f}\right)$, the **average distillate concentration** $\left(x_{D,ave}\right)$ will be calculated.

Objective 2: Use experimental data to predict the time required to obtain a particular concentration of the distillate

A. Boil-up Rate and Reflux Ratio of distillation system using recorded time at total reflux, recorded time at 1/6 opening reflux, and the amount of bottoms at initial and final.
B. The **predicted time of distillation** ($t_{predict}$) was calculated based on Equation by Seader et al. (2016).
C. Calculate % error of time of distillation based on recorded values for actual and predicted.

Objective 3: Determine the total number of stages for the batch distillation process.

A. Obtain weight composition at distillate $\left(x_D\right)$ and weight composition at bottoms (x_B). Also obtain amounts, in mass, for Bottoms and Distillate.
B. Use Fenske Equation to obtain minimum number of steps for fractional batch distillation
C. Use McCabe-Thiele Graphical Method to obtain minimum number of steps for fractional batch distillation.
D. Compare results from both methods and observe key trends and inferences.

Materials, Measuring Apparatus & Equipment

Material	**Quantity**	
Ethanol (analytical grade)	Calibration: 20% w/w, 40% w/w, 60% w/w, 80% w/w, 100% w/w	
Ethanol (technical grade)	30% w/w	
Distilled Water	100% w/w	
Steam	-	
Apparatus	**Size**	**Quantity**
Refractometer	-	1
Volumetric Flasks (Calibration)	50 mL	6
Thermometer	-	1
Steam Pressure Gauge	-	1
Graduated Cylinder	3L	1
Weighing Scale	Max Capacity: 30 kg.	
Equipment	**Specifications**	
Stirring Rod	Large enough for mixing 27L of Ethanol solution	
Container	Volume = 30L	
Beaker or Sample Collecting Vessel	Volume ≈ 50mL	
Batch Distillation Set-up	**Reboiler:** Steam Heated **Heat exchanger:** Boiler type fitted externally to the spherical vessel in a thermosyphon loop **Special Vessel:** 20-L capacity	

Overhead Condenser: Cooling water of inlet temperature at 20°C and condensation temperature of 100°C
Product Receiving Vessels: 5-L capacity
Product Cooler: Generally, gives product temperature less than 10°C above coolant supply temperature.
Auxiliary Apparatus: Thermometer, Steam Pressure Gauge

Task Plan

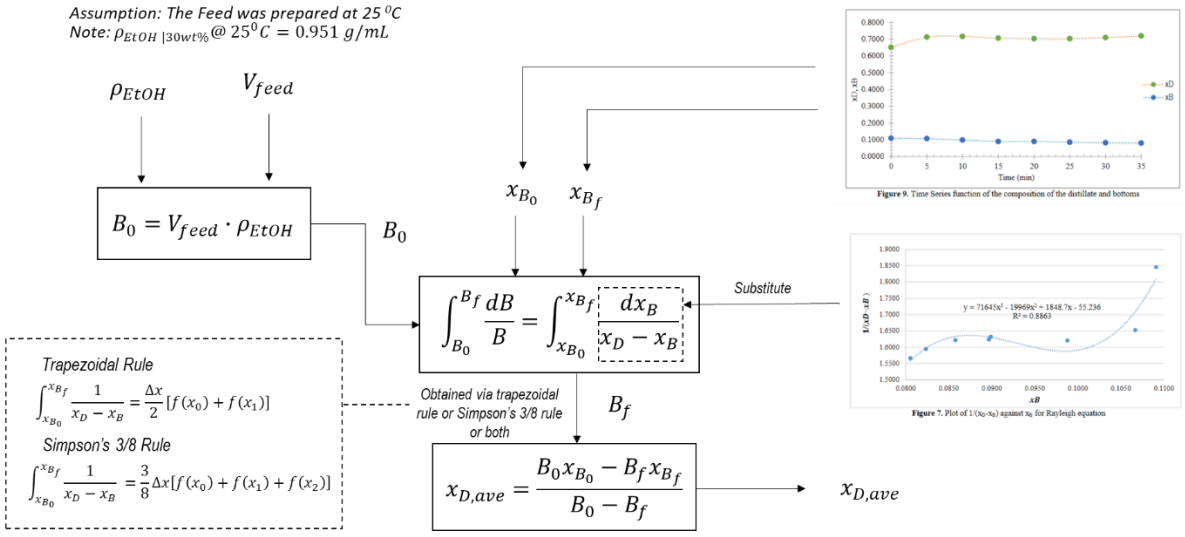
Time	Task	Person Responsible
September 28, 2021		
12:00 – 3:00	Pre-Laboratory Virtual Questioning (thru Google Meet)	Canama Delco Talandron
September 30, 2021 (Calibration Day)		
12:00-2:30	Preparation of ethanol solutions	Canama
	Measuring of refractive indices	Talandron
	Plotting weight concentration vs refractive index	Delco
2:30-3:00	Determination of trendline	Canama Delco Talandron
October 1, 2021		
12:00-12:15	Preparation of 27L of 30% w/w ethanol solution feed	Canama Delco Talandron
12:15-12:45	Open all vents	Canama
	Close drain valve, steam supply valve, recycle valve, reflux control valve, product receivers valve, and soft water inlet valve	Delco
	Fill the still with feed	Delco
	Open the soft water inlet valve	Talandron
	Place thermometers	Canama
	Open then close by-pass valve Open steam supply valve	Talandron
	Adjust regulating valve to 10 psig	Talandron
	Open steam inlet valve	Delco
12:45-1:05	Operate at total reflux for 20 mins	Canama Delco Talandron

1:05-1:15	Open reflux valve Wait for upper product receiver to fill	Talandron
1:15-1:25	Open recycle valve Wait for product to drain	Delco
1:25-1:35	Close recycle valve	Canama
	Operate at total reflux for few minutes Wait for upper product receiver to fill	Talandron
1:35-1:45	Operate at 1/6 reflux	Canama
	Withdraw distillate into 2L graduated cylinder	Delco
1:45-2:20	Take samples of distillate every 5 mins	Canama
	Take samples of still mixture every 5 mins	Talandron
	Measure temperature every 5 mins	Delco
2:20-2:30	Set to total reflux	Talandron
	Close steam regulating valve	Delco
	Vent residual steam	Talandron
2:30-2:40	Turn off condenser cooling water when boiling has stopped	Delco
	Close water supply valve Wait for bottoms to cool	Talandron
2:40-2:50	Drain to suitable receiver	Canama
October 2, 2021		
1:15 – 1:30	Calculations for Average Distillate Concentration	Canama Delco Talandron
1:30 – 1:45	Determination of Predicted Time of Distillation	Canama Delco Talandron (Note: Each member shall do the procedures **individually and simultaneously**)
1:45 – 2:00	Compare predicted time of distillation to actual time of distillation	Canama Delco Talandron
2:00 – 2:15	Collection of Raw Data	Delco
	Collection of Documentations (Screenshot of Raw Data; Proof of Conduct of Experiment)	Canama
2:15 – 2:45	Analysis and Processing of Observed Mechanisms and Data Values from the Simulator	Canama Delco Talandron
2:45 – 2:50	Partial Wrap-up of Discussions	Talandron
October 3, 2021		
12:30 – 2:00	Continuation of Data Process and Analysis	Canama Delco Talandron
	Review of Calculations	
2:00 – 2:45	Finalization of Actual Conduct of Experiment	

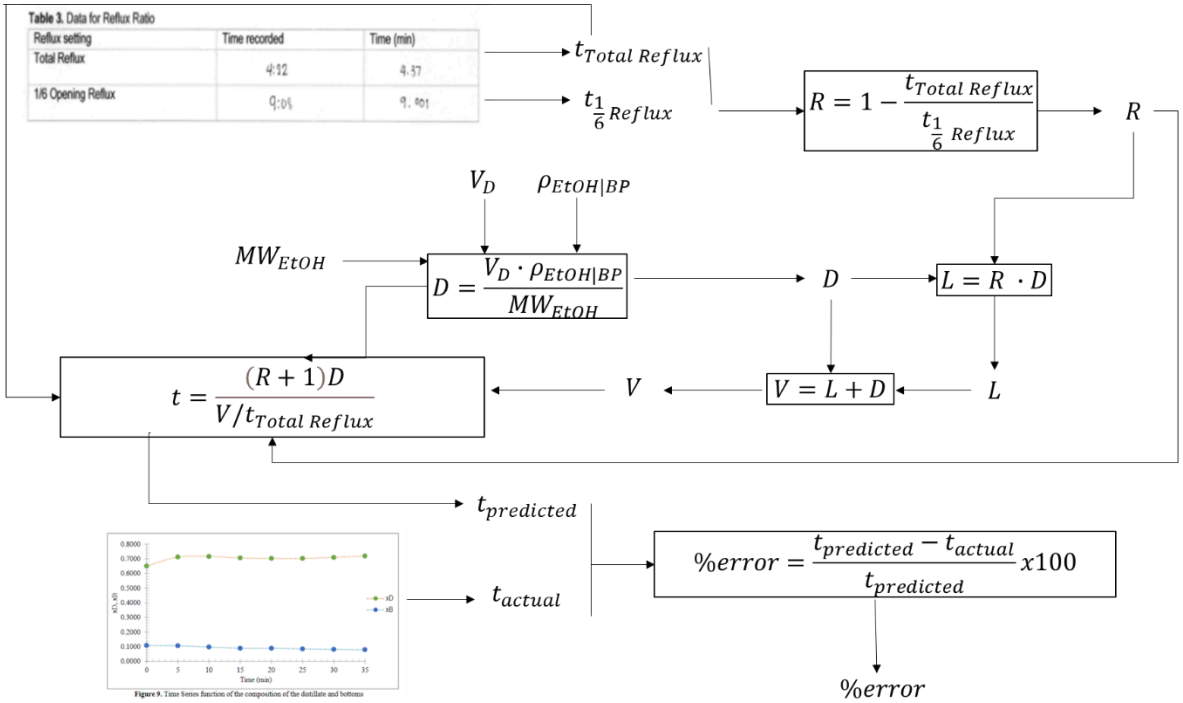
2:45 – 2:50	Submission of Documents	
2:50 – 3:00	Wrap-up of Discussions	

Information Flow Diagram

Objective 1: Determine the average concentration of a specified volume of the distillate that is obtained from a feed mixture of known composition by operating the distillation unit at constant reflux.



Objective 2: Use experimental data to predict the time required to obtain a particular concentration of the distillate.



Objective 3: Determine the total number of stages for the batch distillation process.

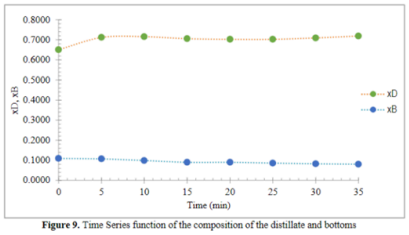


Figure 9. Time Series function of the composition of the distillate and bottoms

$x_{D,HK}$

$x_{D,LK}$

$x_{B,HK}$

$x_{B,LK}$

$$N_{min} = \frac{\ln\left(\frac{x_{D,LK}x_{B,HK}}{x_{B,LK}x_{D,LK}}\right)}{\ln\left(\frac{K_{LK}}{K_{HK}}\right)}$$

N_{min}

$$K_{LK} = \frac{x_{D,LK}}{x_{B,LK}} \quad K_{HK} = \frac{x_{D,HK}}{x_{B,HK}}$$

Legend

Symbol	***Nomenclature (Unit)***
ρ_{EtOH}	Density of 30 wt% Ethanol Solution @25 ^{0} C $(= 0.951\ g/mL)$
V_{feed}	Volume of Feed $(= 27\ L)$
B_0	Initial Mass of Bottoms (kg)
B_f	Final Mass of Bottoms (kg)
x_{B_0}	Initial Composition (mass fraction) at the Bottoms
x_{B_f}	Final Composition (mass fraction) at the Bottoms
$x_{D,HK}$	Composition (Mole Fraction) of Distillate (More Volatile)
$x_{D,LK}$	Composition (Mole Fraction) of Distillate (Less Volatile)
$x_{B,HK}$	Composition (Mole Fraction) of Bottoms (More Volatile)
$x_{B,LK}$	Composition (Mole Fraction) of Bottoms (Less Volatile)
x_D	Composition (Mole Fraction) of Distillate at time t
x_B	Composition (Mole Fraction) of Bottoms at time t
$x_{D,ave}$	Average Composition of Distillate
Δx	Difference between the value of concentration of bottoms at time t and at t+5 (in minutes)
$f(x_i)$	$\frac{1}{X_D - X_B}$ as a function of concentration
$t_{Total\ Reflux}$	Time elapsed to fill upper distillate receiver at Total Reflux (min)
$t_{\frac{1}{6}\ Reflux}$	Time elapsed to fill upper distillate receiver at 1/6 Opening Reflux (min)
$\rho_{EtOH\|BP}$	Density of 30 wt% ethanol at Boiling Point (41.4 ^{0}C) $(\approx 0.941\ g/cm^3)$

MW_{EtOH}	Molecular Weight of Ethanol (g/mol)
R	Reflux Ratio
L	Mole Ethanol Fed Back to the Still (mol)
D	Mole of Distillate (mol)
V	Mole Flowrate of Vapor (mol/min)
$t_{predicted}$	Predicted Distillate Time (min)
t_{actual}	Actual Distillate Time (min)

Raw Data Sheets

Student Performing: Canama

Feed Properties

Volume of Feed (L)	

Data for Reflux Ratio

Reflux Setting	Time (min)
Total Reflux	
1/6 Reflux	

Molar Compositions of Distillate and of Bottoms at Each Time Interval

Time (min)	Distillate Composition	Bottoms Composition
0		
5		
10		
15		
20		
25		
30		
35		

Temperatures

Setting	Temperature (˚C)
Ambient	
Operating	
Boiling Point of 30 wt% EtOH	

Observations

Documentation

Student Performing: Delco

Times for Distillation

Reflux Setting	Time (min)
Total Reflux	
1/6 Reflux	

Compositions of Distillate and of Bottoms at Different Times (every 5 min)

Time (min)	Distillate Composition	Bottoms Composition
0		
5		
10		
15		
20		
25		
30		
35		

Observations

Documentation

Student Performing: Talandron

Feed Composition

Volume of Feed (L)	

Times for Distillation

Reflux Setting	**Time (min)**
Total Reflux	
1/6 Reflux	

Compositions of Distillate and of Bottoms at Different Times (every 5 min)

Time (min)	**Distillate Composition**	**Bottoms Composition**
0		
5		
10		
15		
20		
25		
30		
35		

Temperatures

Setting	**Temperature (˚C)**
Ambient	25
Boiling Point of 30 wt% EtOH	41.4

Observations

Documentation