ST5: Oldershaw Distillation

Group B5
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Contents

1 Introduction

The purpose of this experiment was to gain a better understanding of the distillation principles and the parameters that affects the operation of distillation columns. In the experiment, a mixture of water and ethanol was separated using an Oldershaw distillation column. Samples from the distillate and the bottoms product were analyzed using gas chromatography. There were two main focus points of the experiment: To determine the time required to reach steady state conditions in the distillation column, and determine the efficiency of a column as a function of the vapor velocity.

2 Theory

2.1 The Distillation

In a distillation process, a mixture is physically separated into two or more products with different boiling points. This happens by partial vaporization of a liquid mixture or by a partial condensation of a gas mixture. The liquid phase gets a lower volatility (with a higher boiling point) component, and the vapor phase gets a higher volatility (with a lower boiling point) component.[1]

In Oldershaw distillation, the column is made up of perforated plates with center-to-sides downcomers connecting the trays. A liquid mixture is heated until vaporization. The vapor moves through the small perforations in the trays, and the liquid flows down through the downcomers.[2] This causes the vapor and the liquid to be in contact, which creates an equilibrium on each tray. The vapor leaving the top of the column, is condensed back into liquid using a condenser. Typically, some of the liquid from the condenser will be sent back into the column a reflux. The rest is removed from the column as distillate.[3]

It is possible to run a distillation using a partial or a total condenser. A partial condenser only partially condenses the vapor into liquid. A total condenser condenses all the vapor back into liquid. The reflux is the portion of the vapor that is condensed and later returned to the distillation column.[2] In this lab, the column will mostly be worked with a total reflux.[1] That means that all vapor at the top of the column has been condensed, and is introduced back into the distillation column. In this experiment the column will be operated using a total condenser with total reflux.

The column efficiency in a distillation column is the ratio of the total number of ideal equilibrium stages to the number of trays in the column:

$$\text{Column Efficiency} = \frac{N_t - 1}{N_a} 100\% \tag{2.1.1}$$

In an ideal distillation stage, the outgoing liquid and vapor phase establishes an equilibrium with each other. If the column efficiency is high, it indicates that there is a thorough contact between gas and liquid.

2.2 McCabe-Thiele

One of the easiest and most instructive ways to analyze binary distillations is by using the McCabe-Thiele method. It is a graphical approach presented by McCabe and Thiele in the 1920s[1]. The method is commonly used to determine the number of theoretical stages needed for a distillation.[2]

The method is based on the assumption of constant molar flow, which implies constant molar vapor flow rate, and constant molar liquid flow rate leaving every stage in the section. Therefore the operating line can be considered a straight line in each section of the distillation column.[1]

2.3 Gas Chromatography

Gas chromatography is a separation method for separating chemical compounds in order to analyze them. The method uses a mobile phase, an inert gas, which is carried through a stationary phase, a liquid or a solid. The chemical compound gets dissolved in the mobile phase, which is then sent through the column with the stationary phase. The separation happens because of the difference in the distributions and volatility of the gas phase compounds. The less volatile molecules will interact more with the stationary phase than the more volatile molecules, which will interact more with the mobile phase. This causes the more volatile molecules to pass quicker through the column than the less volatile molecules, which in turn causes the separation. A detector at the end will measure the amount of the compounds after they pass through.[4]

3 Experimental Procedure

The experiment was divided into two parts: Finding the required time to reach steady state, and studying the correlation between column efficiency and vapor velocity.

3.1 Apparatus

The experiment was performed using an Oldershaw distillation column with total reflux. The column had 15 trays, and a reboiler. The reflux was controlled by a computer connected to a pump. The samples taken during the experiment were analyzed using gas chromatography.

3.2 Column Startup

The computer was turned on, and then the cooling water. The bottom and distillate tap lines were closed and the column was filled with a mixture of 1730 mL of ethanol and 4070 mL of water, so that the molar fraction of ethanol in the mixture, x_{EtOH}, was 0.11. Furthermore, the column boiler was set to $T = 105\,^{\circ}\mathrm{C}$. During the experiment, the boiler's power duty was set to different values. The clock was started when the vapor stream started to condensate.

3.3 Exercise 1: Time Required Until Steady State

The meaning of this exercise was to calculate the time it took to reach steady state conditions.

The reboiler was set to 50 %. Within 60 minutes, samples were taken from the top every fifth minute. At the end of the experiment there were 12 top-samples, and a sample from the bottom-column which was taken after the 12th top-sample. All the samples were analyzed in order to figure out how long it took to reach steady state in the column.

3.4 Exercise 2: Efficiency vs. Vapor Velocity

The purpose of this exercise was to find the variation of the column efficiency as a function of the vapor velocity. The reboiler was set to 40% power, and the distillation ran for 45 minutes. After that, 20 mL-samples from both the top and the bottom were taken, and the reflux rate and the temperature in the top of the column was written down.

The experiment was repeated for the 60% and the 70% power setting. In total the column were run using three different boiler power settings during exercise 2.

3.5 Gas Chromatography

Using mikropipettes, 500 µL of the distillation samples, and 250 µL isopropanol were added to a small vials. The vials were then mixed using a table-top shaker, and analyzed using gas chromatography.

4 Results

4.1 Experiment 1

Table 4.1 shows the collected data from the first samples, when the boiler's power duty was set to 50%:

Table 4.1: Data collected during experiment 1, when the boiler's power duty was set to 50%

Sample number	Minutes $[min]$	Concentration $[V\%]$
1	5	83.469
2	10	91.090
3	15	96.479
4	20	99.074
5	25	94.012
6	30	97.261
7	35	96.036
8	40	98.203
9	45	94.204
10	50	93.501
11	55	96.227
12	60	103.989
13 - bottom	60	22.307

Some of the data collected during the experiment were unusable due to the ethanol concentration being more than 96%, which is physically impossible. In order to get better accuracy in finding the time required to reach steady state, it was necessary to aqquire more data points. Table 4.2 shows experimental data collected from group B21.

Table 4.2: Data from experiment 1, collected from group B21

Sample number	Minutes $[min]$	Concentration $[V\%]$
3	15	92.68
11	55	95.36

Figure 4.1.1 shows the data from experiment 1. The red and grey points are from our own experimental data, but the grey points are excluded for being more than 96 V % ethanol, which is not possible. The green points are extra data points collected from group B21, in order to increase the accuracy of the curve fit.

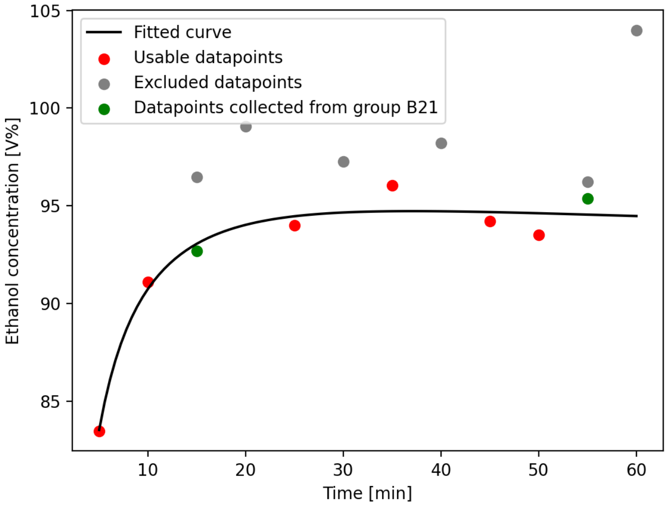

Figure 4.1.1: Volume fraction of ethanol in the distillate as a function of time

4.2 Experiment 2

Table 4.3 show the data collected during experiment 2, with the boiler set to different values. The concentrations are the volumetric percentage of ethanol in the samples. The ethanol concentration in top sample taken during distillation at 40% power duty was larger than 96%, to get more accurate data, experimental data was collected from group B21. The extra data is also presented in the table. The data from 50% power duty was collected from Table 4.1, with the top sample being the last sample having measured an ethanol concentration lower than 96%, which was sample number 10.

Table 4.3: Data collected during experiment 2, with the boiler set to different values. The concentrations are the volumetric percentage of ethanol in the samples. Extra data collected from group B21 is also presented in the table.

Power duty [%]	Concentration [V%]		R_{reflux} [mL/ min]	Temperature [°C]
	Top	Bottom		
40	104.017	22.199	37	76.8
40 - Group B21	92.250	15.425	41	82.4
50	93.501	22.307	54	76.9
60	81.697	22.963	74	76.7
70	92.727	21.654	90	76.8

Table 4.4 shows the relevant values calculated from the data collected during experiment 2. The theoretical number of stages were found using the McCabe-Thiele diagrams in Appendix B.4. All of the calculations are explained in detail in Appendix B. The data from collected from group B21 was used for the 40% power duty setting.

Table 4.4: The calculated values of the molar fractions of the distillate, x_D, the molar fractions of the bottoms, x_B, the vapor velocity, v_{gas}, the theoretical number of stages in the distillation, and the total column efficiency for each power duty used during experiment 2.

Power duty [%]	x_D	x_B	v_{gas} $[m/s]$	Theoretical number of stages	Total column efficiency [%]
40	0.786	0.053	0.103	5.00	26.7
50	0.816	0.081	0.133	6.35	35.7
60	0.579	0.084	0.224	1.83	5.53
70	0.797	0.078	0.225	5.45	29.7

Figure 4.2.1 shows the total column efficiency plotted against the vapor velocity for the different boiler power duties.

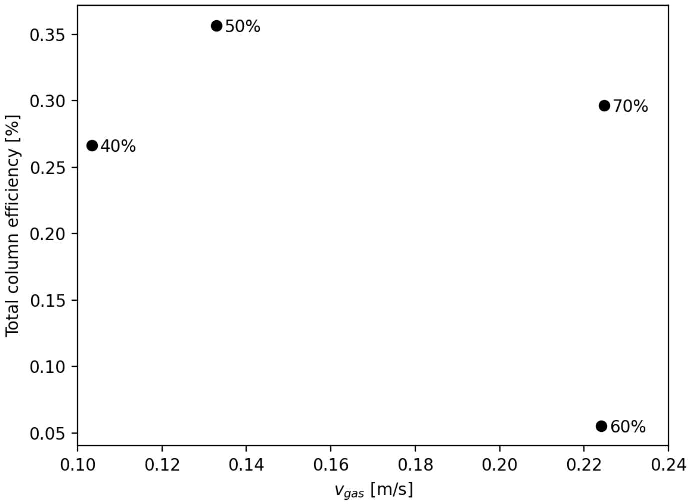

Figure 4.2.1: The total column efficiency plotted as a function of the vapor velocity, for the different boiler bower duties used during the experiment.

5 Discussion

The time the system took to reach steady state for a power level at 50% was estimated from the data in Table 4.1 and is presented in Figure 4.1.1. The volume fraction of ethanol is stabilizing at around 25 minutes according to the fitted curve. However, this is close to one of the values from the other group, and the least accurate values are excluded. When the original points are used, steady state is not reached. Several of the samples had a volume percentage higher than 96%, which is the concentration of the alcohol we added to the column. If the measured values were correct, ethanol would have been produced during this experiment, which is not possible. This means that the measurements must have been of bad quality, with significant errors occurring throughout the experiment.

The data from experiment 2 is also difficult to draw conclusions from. From Figure 4.2.1, the data points are spread out in a fashion that makes it impossible to draw any conclusions about the relationship between the total column efficiency and the vapor velocity. It was observed that increasing the power duty, also increased the reflux rate of the column. In theory, this should have resulted in a cleaner product, and thus a lower total column efficiency, however, the data from the experiment does not show this. It is also expected that increasing the power duty should increase the vaporization in the bottom of the column, which in turn should lead to a somewhat linear increase in vapor velocity, this matches the data, except for the 60% power duty, which most likely is due to a combination of bad reading of the reflux rate, and a bad measurement from the gas chromatography.

5.1 Error Sources

There are several sources of errors in this experiment. It was expected errors from the apparatus from the thermometers, the pump and the sensors, and in addition there was many other errors.

The column was assumed to be working under total reflux. This can not be true since 19 samples of high purity of ethanol were taken out for analysis at different points in the experiment. But the volume of the samples were really small compared to the total volume in the column, therefore, this error is probably not so big that it had any major impact on the results.

Gas chromatography were used to measure the alcohol content, and the apparatus that preformed gas chromatography had an uncertainty of around 1%. Also, since the amounts of liquid that were analyzed were so small, small human mistakes in the pipetting process would have a great impact on the error. The pipetting might have been done too slow, so that some of the liquid evaporated. It is also possible that the samples were not mixed properly. Therefore, the gas chromatography process was a significant source of error, and most likely the biggest reason the collected data does not show the expected trends. To correct this, the samples should have been analyzed again, using more care when preparing the samples. This was not done due to time limitations and equipment failure.

6 Conclusion

In this experiment, distillation principles in an Oldershaw column has been observed. A mixture of ethanole and water was separated, and the samples from the top and bottom products were analyzed using gas chromatography. From the collected data, the time to reach steady state was estimated to be 25 minutes. The data from experiment 2 was inconclusive, and no conclusions about the relationship between the vapor velocity and the total column efficiency could be drawn. The biggest source of error in the experiment was the preparation of the samples that were analyzed using gas chromatography. In order to get better data, the experiment should be repeated, and the samples should be prepared with more care.

Trondheim, January 9, 2022

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References

[1] Heinz Preisig. Felleslab: Distillation columns. *Felleslab*, 2021.

[2] J. D. Seader, E. J. Henley, and D. K. Roper. *Separation Process Principles with Applications Using Process Simulators*. John Wiley & Sons, Hoboken, NJ, 4th edition, 2019.

[3] R. A. Shellie. Distillation column. `https://www.sciencedirect.com/topics/engineering/distillation-column`, 2012, Acessed 22.09.21.

[4] R. A. Shellie. Gas chromatography. `https://www.sciencedirect.com/topics/agricultural-and-biological-sciences/gas-chromatography`, 2013, Acessed 19.09.21.

[5] Wiley. *Aylward, G.H and Finlay, SI Chemical Data 7th edt.* Australia, 2014. ISBN 978-0-7303-0246-9.

[6] J. Gmehling and U. Onken. *Vapor-Liquid Equilibrium Data Collection: Aqueous-Organic Systems (Vol.1, Part 1).* Dechema, Germany, 1977.

A Data Used in Calculations

The relevant physical properties of ethanol and water are presented in Table A.1.

Table A.1: Physical data for ethanol, EtOH, and water, H_2O.[5]

Compound	M [$\mathrm{g\,mol^{-1}}$]	ρ [$\mathrm{g\,mL^{-1}}$]
EtOH	46.068	0.785
H_2O	18.016	0.997

Table A.2 shows the VLE data for ethanol-water mixture at 1 atm:

Table A.2: VLE data for for ethanol-water mixture at 1 atm. Collected from *Vapor-Liquid Equilibrium Data Collection: Aqueous-Organic Systems*[6].

x_{EtOH}	y_{EtOH}
0.000	0.000
0.050	0.337
0.100	0.452
0.150	0.506
0.200	0.536
0.250	0.559
0.300	0.579
0.350	0.599
0.400	0.618
0.450	0.637
0.500	0.656
0.550	0.677
0.600	0.699
0.650	0.725
0.700	0.755
0.750	0.784
0.800	0.817
0.850	0.859
0.900	0.896
0.950	0.947
1.000	1.000

B Calculations

B.1 The Amounts of Ethanol and Water in the Start Solution

The physical properties of ethanol and water are presented in Table A.1.

A 5800 mL mixture of ethanol and water, with molar composition $x_{\mathrm{EtOH}} = 0.11$, is to be prepared. This mixture will be used as the start solution.

The molar amount of ethanol, n_{EtOH}, is given by

$$n_{\mathrm{EtOH}} = \frac{\rho_{\mathrm{EtOH}} V_{\mathrm{EtOH}}}{M_{\mathrm{EtOH}}} \tag{B.1.1}$$

Due to the start solution being a binary mixture, the volume of water, $V_{\mathrm{H_2O}}$, can be expressed as

$$V_{\mathrm{H_2O}} = V_{tot} - V_{\mathrm{EtOH}} \tag{B.1.2}$$

Using this, the molar amount of water, $n_{\mathrm{H_2O}}$, can be expressed using the difference between the total volume and the volume of ethanol:

$$n_{\mathrm{H_2O}} = \frac{\rho_{\mathrm{H_2O}} \left(V_{\mathrm{tot}} - V_{\mathrm{EtOH}}\right)}{M_{\mathrm{EtOH}}} \tag{B.1.3}$$

The molar fraction of ethanol in a solution is given by

$$x_{\mathrm{EtOH}} = \frac{n_{\mathrm{EtOH}}}{n_{tot}} \tag{B.1.4}$$

Combining equations (B.1.1), (B.1.3) and (B.1.4), gives the expression

$$x_{\mathrm{EtOH}} = \frac{\frac{\rho_{\mathrm{EtOH}} V_{\mathrm{EtOH}}}{M_{\mathrm{EtOH}}}}{\frac{\rho_{\mathrm{EtOH}} V_{\mathrm{EtOH}}}{M_{\mathrm{EtOH}}} + \frac{\rho_{\mathrm{H_2O}} (V_{\mathrm{tot}} - V_{\mathrm{EtOH}})}{M_{\mathrm{EtOH}}}} \tag{B.1.5}$$

The only unknown value in this equation is V_{EtOH}. Inserting all known values into the expression, and solving for V_{EtOH} gives $V_{\mathrm{EtOH}} = 1661.3\,\mathrm{mL}$. In the laboratory, a 96 vol.% solution of ethanol will be used to make the start solution. The necessary volume needed will be

$$V_{\mathrm{EtOH},96\,\%} = \frac{V_{\mathrm{EtOH},100\,\%}}{0.96} = 1730.5\,\mathrm{mL}$$

Using equation (B.1.2), the volume of pure water needed is calculated

$$V_{\mathrm{H_2O}} = V_{tot} - V_{\mathrm{EtOH}} = 4069.5\,\mathrm{mL}$$

B.2 Converting Volumetric Fraction to Molar Fraction

The molar fraction of ethanol in the mixture, x_{EtOH}, can be expressed as:

$$x_{\mathrm{EtOH}} = \frac{n_{\mathrm{EtOH}}}{n_{\mathrm{tot}}} = \frac{n_{\mathrm{EtOH}}}{n_{\mathrm{EtOH}} + n_{\mathrm{H_2O}}} \tag{B.2.1}$$

The following equation can be used to convert the volume of a species, i, to the amount of moles:

$$n_i = \frac{\rho_i V_i}{M_i} = \frac{\rho_i x_{V,i} V_{\mathrm{tot}}}{M_i} \tag{B.2.2}$$

Inserting equation (B.2.2) into equation (B.2.1), for the species EtOH and H_2O, gives the expression

$$x_{\mathrm{EtOH}} = \frac{\frac{\rho_{\mathrm{EtOH}} x_{V,\mathrm{EtOH}} V_{\mathrm{tot}}}{M_{\mathrm{EtOH}}}}{\frac{\rho_{\mathrm{EtOH}} x_{V,\mathrm{EtOH}} V_{\mathrm{tot}}}{M_{\mathrm{EtOH}}} + \frac{\rho_{\mathrm{H_2O}} x_{V,\mathrm{H_2O}} V_{\mathrm{tot}}}{M_{\mathrm{H_2O}}}} \tag{B.2.3}$$

By tidying up the expression, and using that for this binary mixture, $x_{V,\mathrm{H_2O}} = 1 - x_{V,\mathrm{EtOH}}$, the final expression becomes:

$$x_{EtOH} = \frac{x_{V,\mathrm{EtOH}} \rho_{EtOH} M_{\mathrm{H_2O}}}{x_{V,\mathrm{EtOH}} \left(\rho_{EtOH} M_{\mathrm{H_2O}} - \rho_{\mathrm{H_2O}} M_{EtOH} \right) + \rho_{\mathrm{H_2O}} M_{EtOH}} \tag{B.2.4}$$

B.2.1 Example Calculation

Inserting the experimental data in Table 4.3 for the top sample at 60% power duty, and the physical data in Table A.1 into equation (B.2.4), gives

$$x_{EtOH} = \frac{0.81697 * 0.785 * 18.016}{0.81697 * (0.785 * 18.016 - 0.997 * 46.068) + 0.997 * 46.068}$$

(The units had to be removed to fit the equation in the document)

Which gives $x_{EtOH} = x_D = 0.579$. The x_D and x_B in Table 4.4 were calculated from the data in Table 4.3 using equation (B.2.4).

B.3 Vapor Velocity

The vapor velocity is calculated by

$$v_{gas} = \frac{\dot{V}_{gas}}{A} \tag{B.3.1}$$

Where A is the cross sectional area of the pipe and $\dot{V}_{gas}$ is the volumetric flow rate of the gas. The cross sectional area is found using

$$A = \frac{\pi}{4} D^2 \tag{B.3.2}$$

Where D is the diameter of the column.

The volumetric gas flow rate can be calculated using the ideal gas law (assuming ideal gas),

$$\dot{V}_{gas} = \frac{\dot{n}_{\text{tot}} R T}{P} \tag{B.3.3}$$

Where R is the molar gas constant, T is the temperature, P is the pressure and $\dot{n}_{\text{tot}}$ is the rate of the total amount of gas molecules, given by.

$$\dot{n}_{\text{tot}} = \dot{n}_{\text{EtOH}} + \dot{n}_{\text{H}_2\text{O}} \tag{B.3.4}$$

Similarly to equation (B.2.2), the molar flow rate of a species i can be expressed using,

$$\dot{n}_i = \frac{\rho_i \dot{V}_i}{M_i} = \frac{\rho_i x_{V,i} \dot{V}_{\text{liquid}}}{M_i} = \frac{\rho_i x_{V,i} R_{\text{reflux}}}{M_i} \tag{B.3.5}$$

The last equality is because the volumetric liquid flow rate, $\dot{V}_{\text{liquid}}$ is equal to the reflux rate, R_{reflux}.

B.3.1 Example Calculation

In this experiment the column is an Oldershaw column with $D = 0.07\,\text{m}$, using equation (B.3.2) the area becomes

$$A = \frac{\pi}{4}(0.07\,\text{m})^2 = 0.00385\,\text{m}^2$$

Which is constant throughout the entire experiment. Using the distillate composition presented in Table 4.3 at 60% power duty, using equation (B.3.5) the molar flow of ethanol is,

$$n_{\text{EtOH}} = \frac{0.785\,\text{g}\,\text{mL}^{-1} \dot{*}\, 0.81697 * 74\,\text{mL}\,\text{min}^{-1}}{46.068\,\text{g}\,\text{mol}^{-1}} = 1.030\,\text{mol}\,\text{min}^{-1}$$

The solution is a binary mixture, meaning that, $x_{V,\text{H}_2\text{O}} = 1 - x_{V,\text{EtOH}} = 1 - 0.81697 = 0.18303$. The molar flow of water is,

$$n_{\text{H}_2\text{O}} = \frac{0.997\,\text{g}\,\text{mL}^{-1} \dot{*}\, 0.18303 * 74\,\text{mL}\,\text{min}^{-1}}{18.016\,\text{g}\,\text{mol}^{-1}} = 0.750\,\text{mol}\,\text{min}^{-1}$$

Adding the molar flow rates together, using equation (B.3.4),

$$\dot{n}_{\text{tot}} = 1.030\,\text{mol}\,\text{min}^{-1} + 0.750\,\text{mol}\,\text{min}^{-1} = 1.780\,\text{mol}\,\text{min}^{-1}$$

The volumetric gas flow rate can be calculated using equation (B.3.3), using the measured temperature $T = 76.7\,°\text{C} = 349.7\,\text{K}$ assuming a constant pressure of $1\,\text{bar} = 10^5\,\text{Pa}$ in the system:

$$\dot{V}_{gas} = \frac{1.780\,\text{mol}\,\text{min}^{-1} * 8.314\,\text{m}^3\,\text{Pa}\,\text{K}^{-1}\,\text{mol}^{-1} * 349.7\,\text{K}}{10^5\,\text{Pa}} = 0.0518\,\text{m}^3\,\text{min}^{-1}$$

Finally, the vapor velocity can be found using equation (B.3.1):

$$v_{gas} = \frac{0.0518\,\text{m}^3\,\text{min}^{-1}}{0.00385\,\text{m}^2} = 13.48\,\text{m}\,\text{min}^{-1} = 0.224\,\text{m}\,\text{s}^{-1}$$

The rest of the vapor velocities were calculated using the same method. The results are presented in Table 4.4.

B.4 Theoretical Number of Stages Using McCabe-Thiele Diagrams

The theoretical number of stages were determined using McCabe-Thiele diagrams, one for each power duty. The equilibrium line was drawn using VLE-data from *Vapor-Liquid Equilibrium Data Collection*[6]. The x_D and x_B used for the different power duties, are presented in Table 4.4.

Figures B.4.1-B.4.4 shows the digitally drawn McCabe-Thiele diagrams for the different power duties. Figure B.4.5 is a hand-drawn McCabe-Thiele diagram for the distillation with 60% boiler power duty.

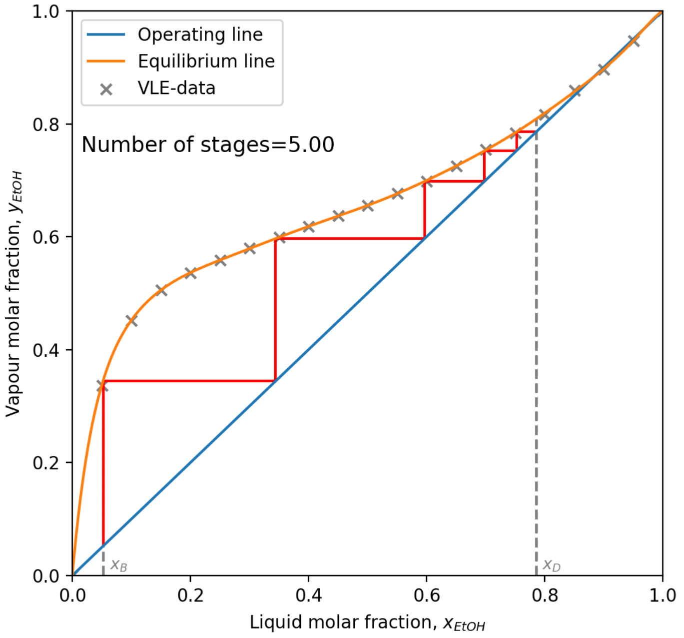


Figure B.4.1: The McCabe-Thiele diagram for the distillation with 40% boiler power duty

B.5 Total column efficiency

The total column efficiency for each power duty was calculated using equation (2.1.1). The number of trays in the column was 15. Using the values from the distillation with 60% boiler power duty, from the McCabe-Thiele diagram, N_t = 1.83. The total column efficiency can then be calculated:

$$\text{Column efficiency} = \frac{1.83 - 1}{15} * 100\% = 5.53$$

The rest of the column efficiencies were calculated similarly. The results are presented in Table 4.4.

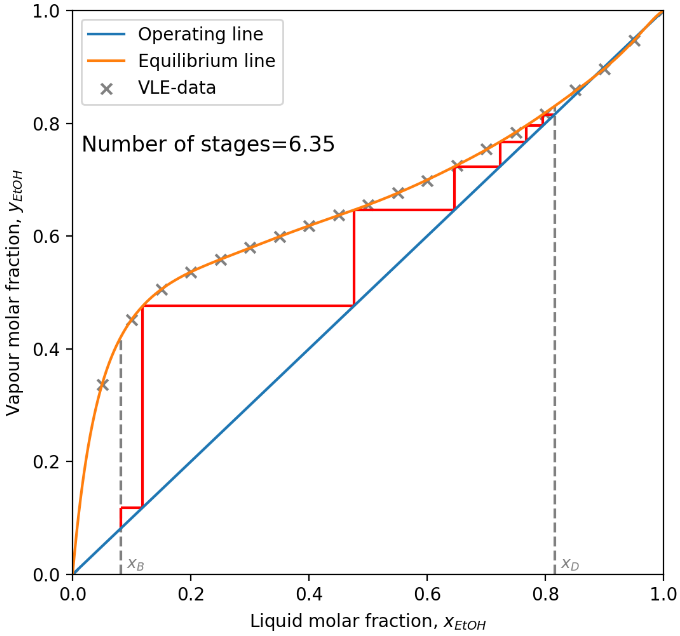


Figure B.4.2: The McCabe-Thiele diagram for the distillation with 50% boiler power duty

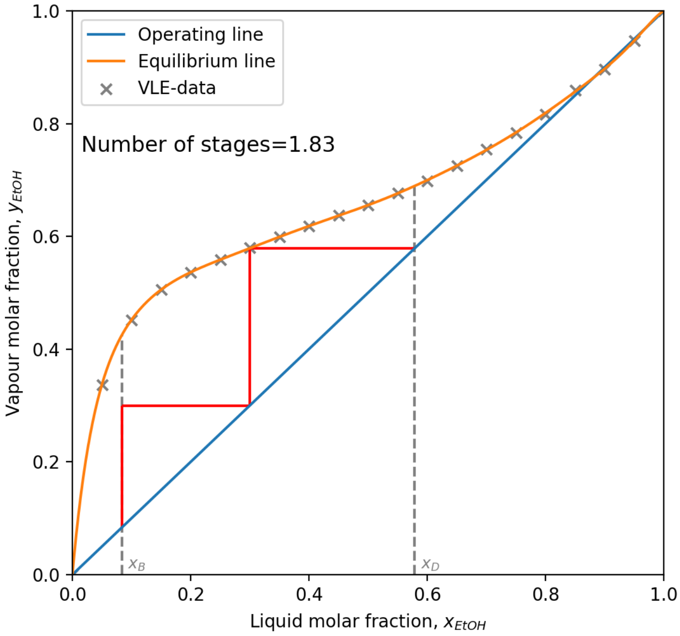


Figure B.4.3: The McCabe-Thiele diagram for the distillation with 60% boiler power duty

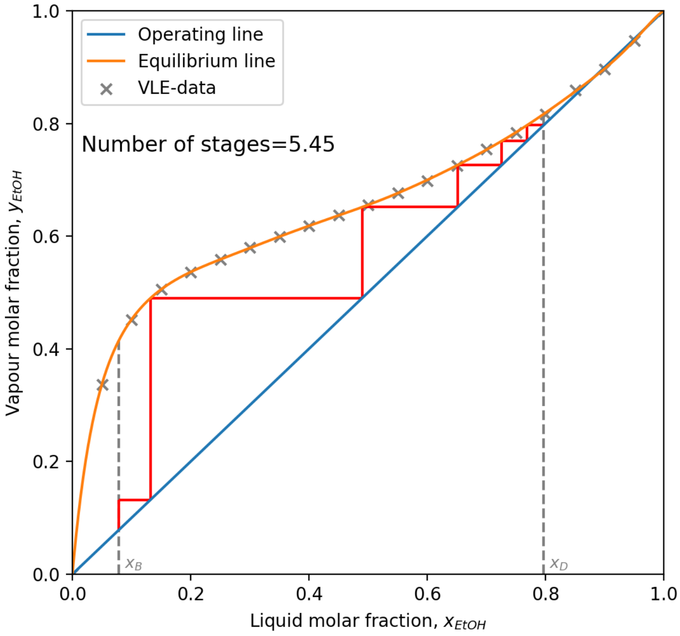


Figure B.4.4: The McCabe-Thiele diagram for the distillation with 70% boiler power duty

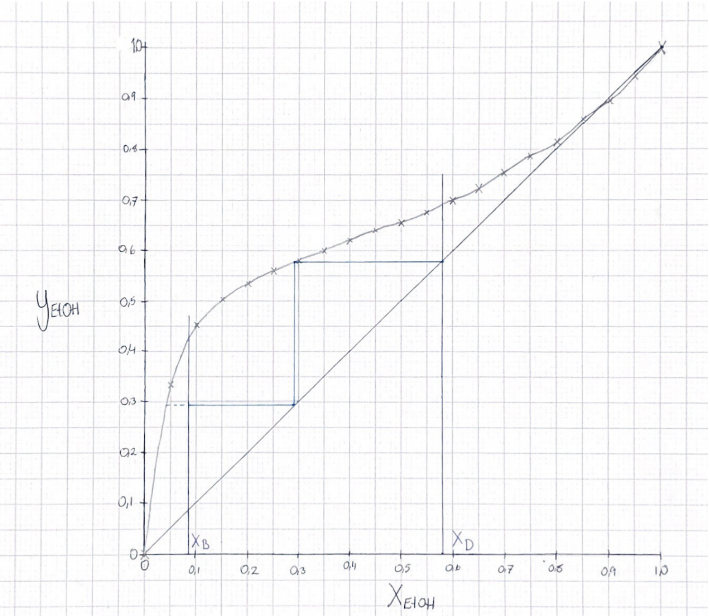


Figure B.4.5: McCabe-Thiele diagram for the distillation with 60% boiler power duty. Made by hand, on a mm-scale paper in A3 format. From the diagram, the theoretical number of steps is estimated to be approximately 1.8.

C Health, Safety and Environment

This was not a high risk experiment, but there are always some potential risk factors involved in an experiment:

Spilling ethanol on a person or an object. Ethanol is not dangerous to humans, and spillage can be easily rinsed off with water. Extra care was taken close to heat-sources, as ethanol is highly flammable. Gloves were used all the times, since ethanol can dry out the skin.

If the equipment was broken or destroyed, it could have caused cuts. In the case of cuts, it would have been necessary to consider how severe the cut was. If it was a small cut, it would need to be cleaned and disinfected.

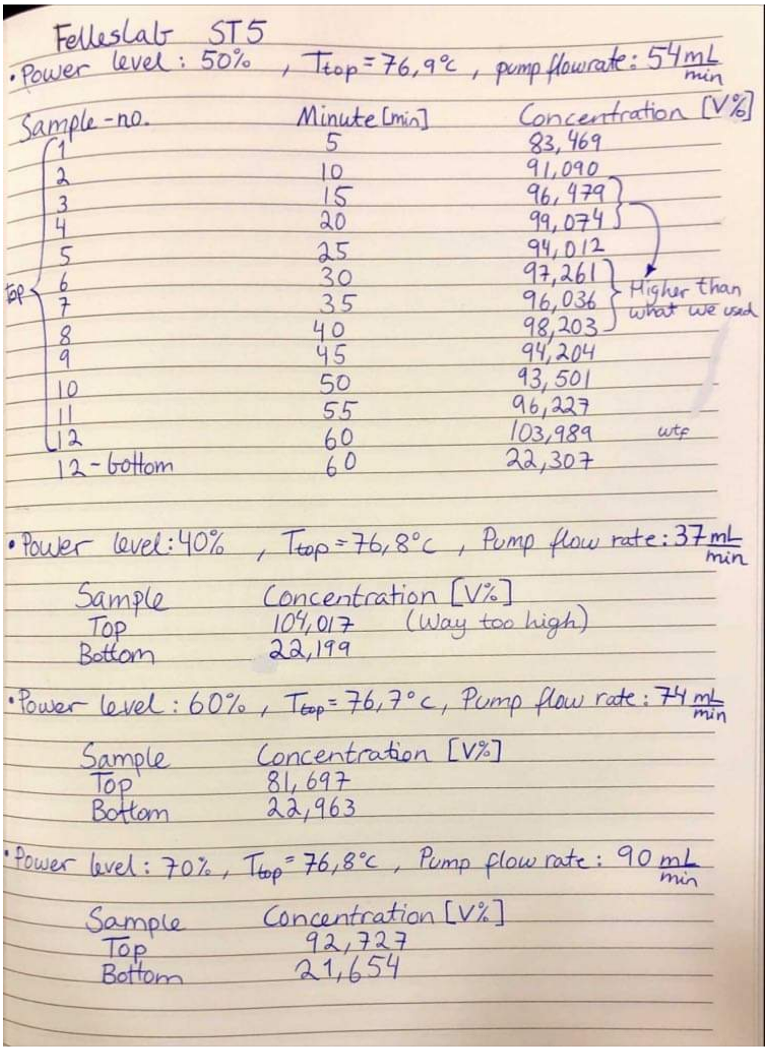
The bottom of the distillation column, the boiler, was kept at a high temperature. Touching it could cause burns. If a burn had occurred, it would have been rinsed with water. Another risk with the high temperature was overflow (flooding) in the column. Then the boiler's heat would have to be turned down.

Leakage from the distillation column was also a risk in this experiment. It was important to always wear personal protective gear (goggles, lab coat and gloves).

Considering COVID-related safety measures, it was important to wash hands when entering and exiting the laboratory, disinfect the work space and all of the equipment and machines that were used, and make sure to keep a distance between each other. Luckily, the COVID restrictions is repealed, so it was not necessary to be strict on the COVID safety measures.

D Lab journal

Figure D.0.1 shows a photocopy of the lab journal written during the experiment.



Felleslab ST5

- Power level: 50%, $T_{top} = 76,9\,°C$, pump flowrate: $54\,\frac{mL}{min}$

	Sample-no.	Minute [min]	Concentration [V%]	
	1	5	83,469	
	2	10	91,090	
	3	15	96,479	
	4	20	99,074	
	5	25	94,012	
Top	6	30	97,261	Higher than what we used
	7	35	96,036	
	8	40	98,203	
	9	45	94,204	
	10	50	93,501	
	11	55	96,227	
	12	60	103,989	wtf
	12-bottom	60	22,307	

- Power level: 40%, $T_{top} = 76,8\,°C$, Pump flow rate: $37\,\frac{mL}{min}$

Sample	Concentration [V%]	
Top	104,017	(Way too high)
Bottom	22,199	

- Power level: 60%, $T_{top} = 76,7\,°C$, Pump flow rate: $74\,\frac{mL}{min}$

Sample	Concentration [V%]
Top	81,697
Bottom	22,963

- Power level: 70%, $T_{top} = 76,8\,°C$, Pump flow rate: $90\,\frac{mL}{min}$

Sample	Concentration [V%]
Top	92,727
Bottom	21,654

Figure D.0.1: Photocopy of the lab journal written during the experiment.

E Python code

E.1 Experiment 1

Below is the python code used to plot the composition of the top samples as a function of time while the power duty was set to 50%.

```
import matplotlib.pyplot as plt
import numpy as np

time = [5, 10, 25, 35, 45, 50]
concentration = [83.469, 91.090, 94.012, 96.036, 94.204, 93.501]
timeerror = [15, 20, 30, 40, 55, 60]
concentrationerror = [96.479, 99.074, 97.261, 98.203, 96.227, 103.989]
additionaltime = [15, 55]
additionalconcentrations = [92.68, 95.36]

timeforfitting = [5, 10, 15, 25, 35, 45, 50, 55]
concentrationforfitting = [83.469, 91.090, 92.68, 94.012, 96.036, 94.204, 93.501, 95.36]

params = np.polyfit(np.log(timeforfitting), concentrationforfitting, 3)
f = lambda x: params[0]*(np.log(x))**3 + params[1]*(np.log(x))**2 + params[2]*(np.log(x)) + params[3]
x = np.linspace(5,60,100)

plt.scatter(time,concentration, color="r", label="Usable datapoints")
plt.scatter(timeerror, concentrationerror, color="grey", label="Excluded datapoints")
plt.scatter(additionaltime, additionalconcentrations, color="g",
            label="Datapoints collected from group B21")
plt.plot(x, f(x), color="black", label="Fitted curve")
plt.xlabel("Time [min]")
plt.ylabel("Ethanol concentration [V%]")
plt.legend()
plt.show()
```

E.2 Experiment 2 - McCabe-Thiele diagrams

Below is the python code used to plot the McCabe-Thiele diagrams.

```
import numpy as np
import matplotlib.pyplot as plt
from pynverse import inversefunc

#Physical data
Mw = 18.016
M_EtOH = 46.068
rho_w = 0.997
rho_EtOH = 0.785

# 40% power duty
#xV_Top = 0.9225
#xV_Btm = 0.15425

# 50% power duty
#xV_Top = 0.93501
#xV_Btm = 0.22307

# 60% power duty
#xV_Top = 0.81697
#xV_Btm = 0.22963

# 70% power duty
xV_Top = 0.92727
xV_Btm = 0.21654


x_D = (xV_Top*rho_EtOH*Mw)/(xV_Top*(rho_EtOH*Mw - rho_w*M_EtOH) + rho_w*M_EtOH)
x_B = (xV_Btm*rho_EtOH*Mw)/(xV_Btm*(rho_EtOH*Mw - rho_w*M_EtOH) + rho_w*M_EtOH)
x_F = x_D

# Getting the equilibrium data, water-ethanol.
# Ethanol fractions
# The function collects the data from the datafiles.
def import_experimental_data(datafile1):
    vle_x, vle_y = np.loadtxt(datafile1, usecols=(0, 1), skiprows=1, unpack=True)
    return vle_x, vle_y


eq_x, eq_y = import_experimental_data("Datafiles/eq_data-ethanol-water.txt")


# This function plots the "stairs" in the McCabe-Thiele diagram. From top to bottom.
def draw_stairs_from_top(x_D, x_B, eq_line_inv, op_line):
    op_line_x_intercept = [x_D]
    opx = op_line_x_intercept[-1]
    opy = x_D
    while eq_line_inv(opy) >= x_B:
        eqx = eq_line_inv(opy)
        eqy = opy
        plt.hlines(eqy, eqx, opx, linestyles="-", colors="r")
        opy = op_line(eqx)
        plt.vlines(eqx, opy, eqy,
                   linestyles="-", colors="r")
        opx = eqx
        op_line_x_intercept.append(opx)
```

```
    eqx = eq_line_inv(opy)
    eqy = opy
    plt.hlines(eqy, x_B, opx, linestyles="-", colors="r")
    plt.vlines(x_B, op_line(x_B), opy,
               linestyles="-", colors="r")
    fraction_of_step = (opx - x_B) / (opx - eqx)
    n = len(op_line_x_intercept) - 1 + fraction_of_step
    return n


# Operating lines, the are the same because we have total reflux
# => R= infinity, q = 1. upper_op_line=lower_op_line=, y=x.
def op_line(x):
    return x


# Creates x-values used in plots
x_values = np.linspace(0, 1, 300)
# Find as function for the eq-line, and its inverse
f = np.poly1d(np.polyfit(eq_x, eq_y, 12))
f_inv = inversefunc(f)

# McCabe-Thiele diagram
fig = plt.figure(figsize=[6, 6])
ax = fig.add_subplot()
plt.plot(x_values, x_values, label="Operating line")
plt.scatter(eq_x, eq_y, marker="x", c="grey", label="VLE-data")
plt.vlines(x_B, 0, f(x_B), linestyles="--", colors="grey")
plt.vlines(x_D, 0, f(x_D), linestyles="--", colors="grey")
plt.plot(x_values, f(x_values), label="Equilibrium line")
number_of_stages = draw_stairs_from_top(x_D, x_B, f_inv, op_line)
plt.xlim(0, 1)
plt.ylim(0, 1)
fig.subplots_adjust(top=0.85)
ax.text(0.015, 0.75, f"Number of stages={number_of_stages:.2f}", fontsize=12)
ax.text((x_B + 0.01), 0.01, r'$x_B$', fontsize=9, c="grey")
ax.text((x_D + 0.01), 0.01, r'$x_D$', fontsize=9, c="grey")
plt.xlabel(r'Liquid molar fraction, $x_{EtOH}$')
plt.ylabel(r'Vapour molar fraction, $y_{EtOH}$')
plt.legend()
#plt.savefig('McCabe-Thiele_diagram.png')
plt.show()
print(f"The number of stages needed are: {number_of_stages:.2f}")
```

E.3 Experiment 2 - Total Column Efficiency vs. Time

Below is the python code used to plot the total column efficiency as a function of the vapor velocity for the different power duties.

```
import numpy as np
import matplotlib.pyplot as plt

# Physical Data
Mw = 18.016
M_EtOH = 46.068
rho_w = 0.997 # g/mL
rho_EtOH = 0.785 #g/mL
pressure = 10**5 #Pa = 1 bar (assumed pressure)
R = 8.314 # m3*Pa/K*mol
Diameter = 0.07 #m

# Experimental Data
boilerpowers = np.array([40, 50, 60, 70]) # %
top_temperatures = np.array([76.8, 76.9, 76.7, 76.8]) + 273 # Celsius, converted to Kelvin
reflux_rates = np.array([41, 54, 74, 90]) # mL/min
xV_top = np.array([0.9225, 0.93501, 0.81697, 0.92727])
xV_btm = np.array([0.15425, 0.22307, 0.22963, 0.21654])
Area = np.pi / 4 * Diameter**2

# Column Efficiency
number_of_theoretical_steps = np.array([5, 6.35, 1.83, 5.45])
number_of_actual_steps = 15
column_efficiencies = (number_of_theoretical_steps - 1)/number_of_actual_steps

# Vapor velocity
n_w = reflux_rates*(1-xV_top)*rho_w/Mw
n_EtOH = reflux_rates*xV_top*rho_EtOH/M_EtOH
n_tot = n_w + n_EtOH # mol/min
volumetric_flow = n_tot*R*top_temperatures/pressure #L/min
v_gas = volumetric_flow/(Area*60) # m/s

fig, ax = plt.subplots()
ax.scatter(v_gas, column_efficiencies, color='black')
for i, power in enumerate(boilerpowers):
    ax.annotate(f"{power}%", (v_gas[i], column_efficiencies[i]),
                textcoords='offset points', xytext=(5,-4) )
plt.xlim(0.1, 0.24)
plt.xlabel(r'$v_{gas}$ [m/s]')
plt.ylabel(r'Total column efficiency [%]')
plt.show()

for i, power in enumerate(boilerpowers):
    print(f"For a power duty of {power}%, v_gas = {v_gas[i]:.3f} m/s and "
          f"the column efficiency is {column_efficiencies[i]*100:.2f}%")
```