

Distillation Column Optimization

CE20238 Chemical Engineering Skills, Practice and Design 2

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Introduction

The aim of this experiment is to calculate the number of theoretical plates required by the column to have the distillation of the ethanol solution from the mixture it is in, which will only be a mixture of ethanol and water. This will be done via a distillation column, in which the mixture of the immiscible liquids will be heated and the ethanol collected in the distillate to a high a mole fraction as possible, and the water be collected at the reboiler, with again, a high mole fraction as possible.

Distillation is a widely used process in industry in purification of substances that are used in the daily life, such as the purification of ethanol in the production of alcoholic drinks. The aim of the process of distillation is to achieve the maximum distillation for the purest component, using the least amount of required plates within the column. This is to achieve maximum efficiency for the process to carry out at the expected yield, with the least possible input.

The basic principle of distillation is that the 2 immiscible liquids are in a solution and need to be separated from one another, in which this problem is overcome by using the different boiling rates as an advantage. The components having different boiling rates means that the temperature of the column can be set in between the 2 boiling temperatures of the components, possibly being closer to the boiling temperature of the MVC (More Volatile Component). This then allows for the MVC to be in the gaseous phase, where the gas will rise and be directed to a storage tank. However, before the storage tank, it is cooler by passing through a cooler to be returned to its liquid state, which makes it safer and easier to store the MVC. The LVC (Less Volatile Component) will remain in the liquid state throughout all this however there will always be slight impurities in the MVC being collected and ultimately this is the challenge that is being faced, to have the least amount of contamination within the MVC by having maximum efficiency in the distillation column.

The first problem to face is the average relative volatility of the mixture, in which the average volatility of the mixture in both the distillate and the reboiler are calculated and the average between the 2 are taken as a representative of the whole distillation column, for this Equation 1 will be used as shown below:

$$(\alpha_{AB})_{avg} = \sqrt{(\alpha_{AB})_d \times (\alpha_{AB})_b} \quad \text{Eq.1}$$

Another problem to face will be the amount of theoretical plates needed within the column, for this, the results obtained from Equation 1 will be used and this will be calculated via the Fenske equation, Equation 2, as displayed below:

$$n_{min} + 1 = \frac{\log \left(\left(\frac{x_A}{x_B} \right)_d \times \left(\frac{x_B}{x_A} \right)_b \right)}{\log (\alpha_{AB})_{avg}} \quad \text{Eq.2}$$

Where the variables that are in the equation equate to:

- n_{min} = Theoretical number of plates required
- x_A = mole fraction of the MVC
- x_B = mole fraction of the LVC
- $(\alpha_{AB})_{avg}$ = average relative volatility of the mixture
- d and b are the subscripts used to describe the conditions in the distillate or boiler, respectively.

Once the minimum number of plates are calculated, the last thing to calculate the column efficiency, for the, the n_{min} value will be used and then rounded up to the next integer so that we have the

minimum amount of plates that can absolutely be used. For this, a percentage will be enough to represent the efficiency of the whole column and this will be done using Equation 3 below:

$$E = \frac{\text{Theoretical number of plates required}}{\text{Actual number of plates in the column}} \quad \text{Eq.3}$$

The efficiency of the distillation column is basically a ratio between the ‘ideal’ number of plates needed for the optimum purification process for ethanol, to the actual number of 8 plates.

Results and Calculations

The initial calculation that was made was the average of the prepared mixtures of the ethanol and water for the calibration curve, in which these values were then used to plot the curve mentioned, which will be refractive index against the volume percentage (vol%), the calculation for the 10% volume ethanol. Further results can be seen in Appendix 1:

$$1.3403 + 1.3401 + 1.3400 = 1.340133$$

Once all of the values for the refractive index were calculated, the next step was to convert the volume percentage to mole percentages, this was done by:

$$\text{For ethanol: } 10\% = 0.1 \text{ ml} = 0.0001 \text{ l} = 10^{-7} \text{ m}^3$$

$$\therefore 10^{-7} \times 798 = 7.98 \times 10^{-5} \text{ kg} = 0.0798 \text{ g}$$

$$\therefore \frac{0.0798 \text{ g}}{46.07 \text{ g mol}^{-1}} = 0.001732 \text{ moles}$$

This was repeated for every vol% of ethanol and water using the density and molecular mass (*NIST - Ethanol*, 2021) for all the amount of moles and then the mole percentages were calculated via the equation below where the mole percentage was converted from 10% volume of ethanol:

$$\frac{\text{No. of moles of Ethanol}}{(\text{No. of moles of water} + \text{No. of moles of ethanol})} \times 100 = \frac{0.001732}{(0.001732 + 0.05)} \times 100 = 3.34\%$$

The calibration curve was then plotted with a line of best fit, which would later be used for the calculation of the mole percentage of the distillate and reboiler values.

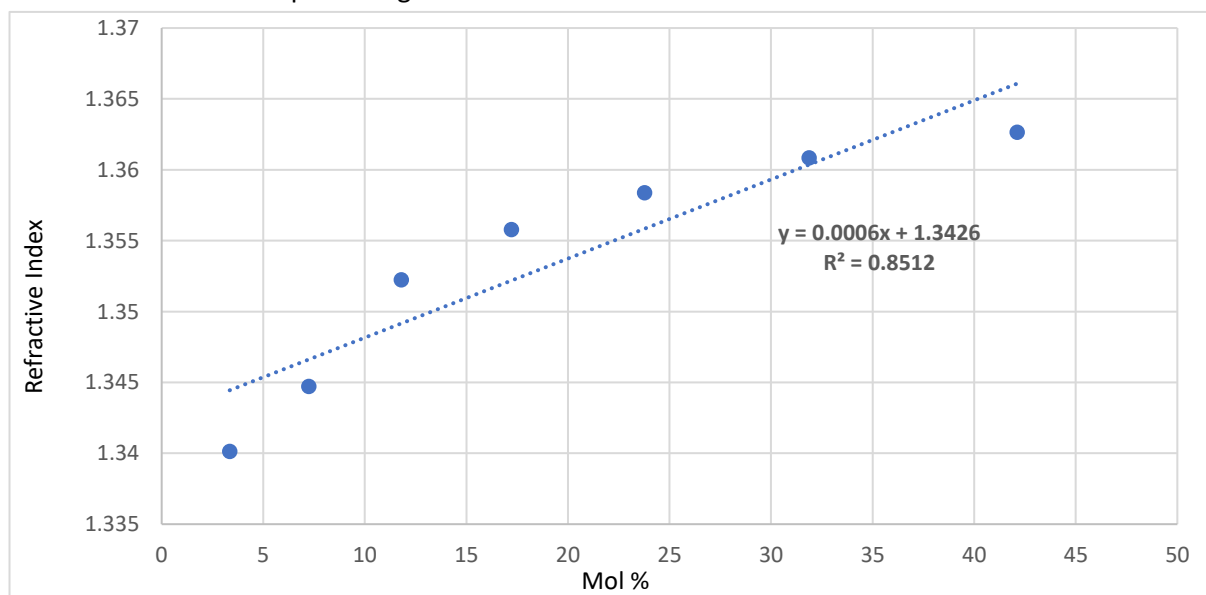


Figure 1: Calibration curve for Refractive Index against the mole percentage of ethanol

When looking at Figure 1, one can see that there is a levelling off of the results almost like the behaviour of a natural log graph, therefore a straight line of best fit was not the best approach to this relationships between the 2 plotted variables, a good solution to this would be to give an exponential line of best fit as it would have a higher chance of being closer to all the point plotted, hence decreasing the R^2 value to give us more accurate mol% values to use when calculating them for ethanol in the reboiler and distillate mixtures.

Once the calibration curve was ready, the values of the mole percentages of ethanol and water could be calculated, for both the distillate and the reboiler mixtures. This was done by using the line of best fit equation in Figure 1, where the x-value represents x_A in Equation 2 and y-value represents the refractive index of our collected solutions, the calculations below will be for the column at 0.6kW of power and the mole fractions will be from the reboiler sample:

$$1.3551 = 0.0006x + 1.3462$$

$$\frac{1.3551 - 1.3462}{0.0006} = 69.3 \therefore x_A = 0.208$$

Once the mole fraction of the ethanol is found, water can be calculated via:

$$1 - x_A = x_B = 1 - 0.2083 = 0.792$$

The next step was to calculate the relative volatilities in both the reboiler and the distillate to have all variable necessary for Equation 1, this was done by finding the Antoine coefficients (Yaws, 2015) which can be found in Appendix 5 and proceeding to use the Antoine's Equation (Thomson, 1946) and a combination of Dalton's and Raoult's law, to use Equation 4, which can be found in Appendix 6:

$$\log(P_A^*) = 5.24677 - \frac{1598.673}{(-46.424 + 365.45)}$$

$$\therefore P_A^* = 1.231 \text{ Pa}$$

In which these values were then used to calculate the relative volatility of the reboiler:

$$(\alpha_{AB})_b = \frac{P_A^*}{P_B^*} = \frac{1.231}{0.4348} = 2.277$$

The results for all the tested scenarios can be found in the Appendix 3. The next step was to start calculating the minimum number of theoretical variables, using Equation 2:

$$n_{min} = \frac{\log\left(\left(\frac{0.693}{0.307}\right) \times \left(\frac{0.792}{0.208}\right)\right)}{\log(2.539)} = 0.443$$

The final step was to calculate the efficiency of the column using Equation 3:

$$E = \frac{0.443}{8} = 5.53\%$$

Another graph that was plotted was the log-log graph for the Pressure difference against the boil-up rate, where the pressure difference was just read off of the manometer and then converted from the units of mmH₂O to Pascals (*mmH₂O to Pa (pascals) Conversion Table*, 2021), the value used was from 0.6kW power distillation, using the method shown below:

$$Pa = mmH_2O \times g$$

$$\therefore Pa = 30\text{mmH}_2\text{O} \times 9.81 = 294.3 Pa$$

As well as this, the boil-up rate also had to be calculated, in which the time taken to fill up a volume of 40ml was taken to calculate a volumetric flowrate value, which can be seen below:

$$\dot{V} = \frac{\text{Volume}}{\text{Time}} = \frac{40\text{ml}}{43.44\text{s}} = 0.921\text{ml s}^{-1}$$

All the other values calculated, including the raw data, can be found in Appendix 7.

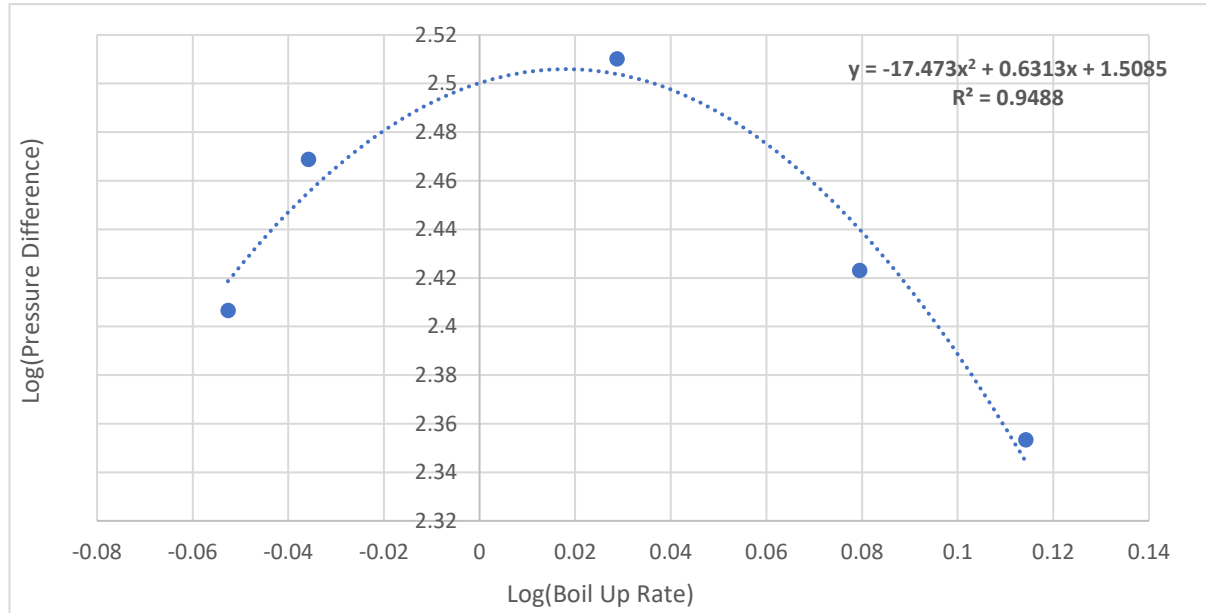


Figure 2: Log-Log Plot for the Pressure Difference in Reboiler and Distillate against the Boil-up rate

Figure 2 shows a parabolic relationship, especially when looking at the equation of the line of best fit as the best way to see the relationship was by looking at the shape of this curve. The pressure difference seems to be peaking at around 2.51 and then decreasing at a faster rate than it increased

References

- *mmH₂O to Pa (pascals) Conversion Table*, 2021. [Online]. S1 Tools. Available from: <https://www.sensorsone.com/mmh2o-to-pa-conversion-table/> [Accessed 3 November 2021].
- *NIST - Ethanol*, 2021. [Online]. Available from: <https://webbook.nist.gov/cgi/cbook.cgi?Name=ethanol&Units=SI> [Accessed 3 November 2021].
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- Yaws, C.L., 2015. *The Yaws Handbook of Vapor Pressure* [Online]. Elsevier. Available from: <https://doi.org/10.1016/c2014-0-03590-3> [Accessed 2 November 2021].

Appendix

1)Table of average values and the standard deviations of the collected Refractive index values for the calibration curve from 0.6 to 1 kW Top to bottom, respectively:

EtOH vol%	Reading 1	Reading 2	Reading 3	STDEV
0	1.3333	1.3331	1.3331	0.000115
10	1.3403	1.3401	1.3400	0.000153
20	1.3445	1.3448	1.3448	0.000173
30	1.3524	1.3522	1.3521	0.000153
40	1.3557	1.3557	1.3559	0.000115
50	1.3584	1.3583	1.3584	5.77E-05
60	1.3609	1.3609	1.3607	0.000115
70	1.3626	1.3627	1.3626	5.77E-05

2)Table of the ethanol and water to moles for mol% from 0.6 to 1 kW Top to bottom, respectively:

ETHANOL

volume(m3)	mass(kg)	mass(g)	moles
0.0000001	0.0000798	0.0798	0.001732147
0.0000002	0.0001596	0.1596	0.003464293
0.0000003	0.0002394	0.2394	0.00519644
0.0000004	0.0003192	0.3192	0.006928587
0.0000005	0.000399	0.399	0.008660734
0.0000006	0.0004788	0.4788	0.01039288
0.0000007	0.0005586	0.5586	0.012125027

WATER

volume(m3)	mass(kg)	mass(g)	moles
0.0000009	0.0009	0.9	0.05
0.0000008	0.0008	0.8	0.044444
0.0000007	0.0007	0.7	0.038889
0.0000006	0.0006	0.6	0.033333
0.0000005	0.0005	0.5	0.027778
0.0000004	0.0004	0.4	0.022222
0.0000003	0.0003	0.3	0.016667

3)Table of the relative volatility values calculated from 0.6 to 1 kW Top to bottom, respectively:

(alphaAB)b	(alphaAB)d	(alpha)avg
2.276569	2.830613	2.53852
2.27596	2.911613	2.57424
2.274441	3.004673	2.614183
2.273835	2.990442	2.607637
2.273835	2.90565	2.570402

4)The calculated pressure and boil-up rate values from 0.6 to 1 kW Top to bottom, respectively:

Boil-up Rate (ml/s)	Pressure Drop (Pa)
-0.03583	2.46879
-0.05264	2.406642
0.028724	2.510183
0.079485	2.423033
0.114215	2.353397

5)Antoine's Coefficients for Ethanol and Water:

	Ethanol	Water
A	5.24677	5.08354
B	1598.673	1663.125
C	-46.424	-45.622

6)Antoine's Equation to find saturated pressure:

$$\log(P_A^*) = A - \frac{B}{(C + T)}$$

7)Values for the calculated plot data for Figure 2, from 0.6 to 1 kW Top to bottom, respectively:

Boil-up Rate (ml/s)	Pressure Drop (Pa)
-0.03583	2.46879
-0.05264	2.406642
0.028724	2.510183
0.079485	2.423033
0.114215	2.353397

8)Values for the calculated plot data for Figure 1, from 0.6 to 1 kW Top to bottom, respectively:

Mol%	Average RI
3.348299	1.340133
7.231026	1.3447
11.78723	1.352233
17.20878	1.355767
23.76808	1.358367
31.86524	1.360833
42.11293	1.362633

9) Values for the density and molecular mass of ethanol and water

	EtOH	Water
Mr	46.07	18
density	798	1000

10) Mole fraction and pressure values calculated for all the collected samples for all powers of distillation.

Power (kW)	Plates	(x _a) _d	(x _w) _d	(x _a) _b	(x _w) _b	(P _a) _b	(P _w) _b	(P _a) _d	(P _w) _d
0.6	8	0.693333	0.306667	0.208333	0.791667	1.230815	0.540645	1.230815	0.434822811
0.7	8	0.603333	0.396667	0.251667	0.748333	1.240273	0.544945	1.240273	0.425974507
0.8	8	0.611667	0.388333	0.208333	0.791667	1.264182	0.555821	1.264182	0.420738558
0.9	8	0.628333	0.371667	0.203333	0.796667	1.273852	0.560222	1.273852	0.425974507
1	8	0.633333	0.366667	0.193333	0.806667	1.273852	0.560222	1.273852	0.438405126

11) The values for the minimum amount of theoretical plates and column efficiencies, from 0.6 to 1 kW Top to bottom, respectively:

n(min)	Column Efficiency
0.44261	5.53266
0.29103	3.637878
0.08354	1.044197
0.12304	1.538015
0.06579	0.822396

12) All the raw data from the distillation column mixtures:

Power (kW)	Reboiler RI	Distillate RI	Pressure Drop (mmH ₂ O)	T(bottom) (C)	T(top) (C)	Volume (ml)	Time (s)
0.6	1.3551	1.361	30	356.45	351.05	40	43.44
0.7	1.3577	1.3664	26	356.65	350.55	35	39.51
0.8	1.3551	1.3659	33	357.15	350.25	40	37.44
0.9	1.3548	1.3649	27	357.35	350.55	40	33.31
1	1.3542	1.3646	23	357.35	351.25	40	30.75