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STUDY AND OPTIMIZATION OF THE DISTILLATION PROCESS AT ACQUAVITE S.p.A.

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Abstract

This thesis is about a study conducting during an internship period at Acquavite S.p.A., a grappa production establishment located in Visná di Vazzola.

Firstly, a brief introduction about grappa is given, followed by a description of Acquavite S.p.A.'s story and their current production plant.

Next, the internship's topics are discussed: The quantities of the components in each stream are calculated in order to evaluate the efficiency of the distillation columns available at the plant and the energy duties of the reboilers, the condensers and the heat exchangers.

Lastly, two possible improvements to the distillation train are discussed: the use of a partial condenser in order to increase the energy efficiency of the plant by reducing the used utilities, and the design of a column to recover ethanol from the wastes stream.

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Foreword

This thesis is the result of a six-months long internship at Acquavite S.p.A. located in Visná di Vazzola, from October 2023 to March 2024. This thesis includes five chapters, the first one being an overview on grappa from a historical, chemical and operational point of view. The second one is a brief description of the company, highlighting its story and its present production plant. The following chapters focus on the internship's main topic, with the third chapter being dedicated to data collection and the use of mass balances to calculate the missing data needed for the following steps. The fourth chapter is about the study of the present plant at Acquavite S.p.A., calculating the efficiency of the columns in average working conditions and their energy duties. Lastly, the fifth chapter provides two ideas to improve the plant's production process.

I would like to express my gratitude to Mr. Roberto Castagner for allowing the internship to take place at his facility and to Mr. Roberto Sonego for the help and for the patience shown during the time spent at Acquavite S.p.A. I would also like to express my thanks to Prof. Sara Spilimbergo and Prof. Alberto Bertucco for the many advices received from them and for guiding me through the process of producing and writing this thesis.

Chapter 1 - Introduction

1.1 What is Grappa?

Grappa is the name given to the distillate of fermented pomace, the skin of the grape left after the wine-making process, made from grapes coming exclusively from Italy or from certain areas of Switzerland and distilled exclusively in Italy[1]. If any of those steps are missing, the product has to be called *acquavite di vinaccia* instead. Chemically speaking, grappa is a mixture of water and ethanol, with other components, such as butanol and propanol, making up a very small percentage but being also the main contributors for the taste and scent.

By Italian law, grappa's ethanol's to be at least 37.5% in volume and no more than 86% [1]. Typically, commercial grappa's alcohol content ranges between 40% to 60%. Aside from classifying grappa by its alcohol content, other distinctions can be made based on the type of pomace used (*grappa monovitigno* is used for grappa whose pomace comes from one grape variety only, while *grappa plurivitigno* is the opposite), on its conservation before being bottled up (*giovane* is used for grappa that has been kept in steel or glass containers, *invecchiata* means it has spent at least 12 months in wooden barrels, *riserva* or *stravecchia* are synonyms and correspond to at least 18 months of aging in wooden barrels and lastly, *barricata* is used for grappa that spent at least half of the aging process within specific wooden barrels called *barrique*) And whether herbs and or fruits have been added to obtain a more complex flavoring (*A*romatizzata).[2]

In general, the process to produce grappa is split in four main steps:

1. Pomace fermentation
2. Alcohol extraction
3. Distillation
4. Aging

1.1.1 Pomace fermentation

Grapes by themselves have very little to no ethanol. As such, in order to be able to use pomace as the starting point for the process of grappa production, it's necessary to allow for fermentation to happen to turn the sugars inside the pomace into ethanol. In the past, the process was mainly taking place within bags, small containers or even open-air cement vats. As grappa became more widely requested and the demand for high-quality product increased, the usage of silos where CO_2 can be inserted to reduce the amount of ethanol being decomposed into methanol started becoming more widely used. In the 90s, thanks to a patent from Roberto Castagner, a new method has been developed and has been showing the best result: machines are used to fill plastic tubes holding up to 300 tonnes of pomace each, spraying an acid solution over the pomace and adding selected yeast strains in order to control the fermentation. CO_2 produced durign the fermentation is used to pressurize the tubes, creating an environment hard for bacteria to prosper into, thus allowing for the pomace to be kept for months and for a lower amount of methanol to be produced.[3]

1.1.2 Alcohol extraction

Once the fermentation has reached the desired point, the pomace goes through an alcohol extractor. Saturated steam moving counter-current to the pomace is used to remove the ethanol along with other compounds of varying relevance within the pomace.

1.1.3 Distillation

Distillation is the most important part of the process. Many of the compounds coming from the alcohol extractor have similar boiling points and some of them have unwanted organoleptic properties or are even toxic for humans. In the past, when alembics were used, it was up to the Master Distiller to recognize when the lower-boiling unwanted products (so-called *teste* or heads) had been distilled and the desired product started to boil, and when the most aromatic part of the grappa (*cuore* or heart) was over and the unwanted higher-boiling compounds (*code* or tails) were about to evaporate. Nowadays, with the need for higher volumes of product, alembics are not used anymore, leaving room for distillation columns. At least two of them are necessary for grappa production: the first one to remove water and higher-boiling components from the mixture as the residue, while the second one, whose feed is the distillate of the first column, separates the *cuore* of the grappa from the *teste*, respectively as residue and distillate.

1.1.4 Aging

Once the grappa is collected outside the distillation columns, it is ready to be stored, either in steel drums or directly in wooden barrels, depending on the desired aging process. Usually, grappa right outside the columns is available at around 80% alcohol content, and is stored as such in order to lower the need for storage space. Before being bottled, osmotized water is used to dilute the alcohol to obtain grappa at the desired alcohol content.

1.2 History of grappa

According to Synesius of Cyrene, who lived in the IV century b.C., the first traces of knowledge regarding distillation date back to seven hundred years before Christ in some areas of Egypt, China and Persia, where wine and cider were the preferred starting material to produce distillates. As the Arabs conquered Egypt centuries later, the art of distillation was perfected and spread to Europe, where small monasteries and a handful of alchemists were the only ones dedicated to the study of this topic.

It wasn't until the Middle Ages that distillation started becoming a topic of interest in Italy. Arnoldo da Villanova (1250-1314), Raimondo Lullo (1235-1315) and Michele Savonarola da Padova (1384-1462) wrote papers about distillation, focusing on the production of acquaviti. The latter's *"De arte conficendi aquam vitae semplicem et compositam"* was an especially important work, as it explained how to reach high alcohol concentrations by cooling the top of the alembic. Leonardo da Vinci (1452-1519) had some interest in the topic as well, designing some alembics whose main point was to separate as many of the volatile compounds as possible.

In the 17^{th} century, the *Associazione degli Acquavitai* was born in Venice, a corporation that aimed at studying and passing on the methods of distillation and a way to regulate the production of spirit drinks by requiring a license to produce them. In addition, for the first time, pomace is mentioned in literature as the main ingredient to produce acquavite in a work by Atanasio Kircher dated 1636.

1813 is an important date for distillation. In this year, Baglioni designed the first distillation column as we know them today: a vessel containing trays where vapor and liquid meet each other and exchange mass and energy, something that would become the backbone of industrial distillation.[4]

Despite the studies by many important figures in the wine-making industry (G.B. Cerletti, Matteo Da Ponte, Emilio Barbet, Giorgio Meloni just to name some), grappa kept being widely considered as a drink targeted towards the masses, because of its high alcohol content, simplicity and the presence, often times, of bad-tasting compounds. However, as society recovered after World War II, the technological development allowed to improve the quality of distillates, grappa in particular, making it a more dignified drink that keeps adapting even today to the tastes of society.

1.3 Technical constraints

The production of grappa presents two main challenges: the first one is the necessity to remove methanol from the final product, the second one is the presence of azeotropes between the compounds.

1.3.1 Methanol separation

Methanol is the simplest alcohol. It's a byproduct of alcoholic fermentation, but it's extremely toxic for humans, causing permanent blindness and even death if ingested[5] due to being converted first into formaldehyde and then into formic acid inside the liver. As such, its presence in the final product is heavily monitored and regulated. Italian law sets its upper limit at 1000 g of methanol per 100 liters of pure (100% volumetric) alcohol[1]. It is thus necessary to remove as much as possible from the product.

Considering that ethanol's boiling temperature is 78.4°C and methanol's is 64.7°C, the most efficient way to separate methanol is to withdraw it from the top of the second column, together with the rest of the heads.

1.3.2 Azeotropes

Phlegm (the mixture coming from the alcohol extractor and entering the first distillation column) is made of about a dozen different compounds, among which the ones that are going to be taken into consideration are Acetaldehyde, Ethyl-acetate, 2-Butanol, 1-Butanol, Isobutanol, Propanol, Methyl-butanol, Ethyl-lactate, Hexanol, Methanol, Ethanol and water. It is inevitable that, with so many compounds interacting, some of them form azeotropes. According to Aspen+ data, the pairs forming azeotropes are: Ethyl-acetate and Methanol, Ethyl-acetate and Ethanol, Ethyl-acetate and Water, 2-Butanol and Water, Propanol and Water, Isobutanol and Water, 1-Butanol and Water, Methyl-butanol and Water, Hexanol and Water and, lastly, the more famous and more important for the production of grappa, Ethanol and Water. In Figure 1 are reported the equilibrium curves of the pairings who show an azeotrope.

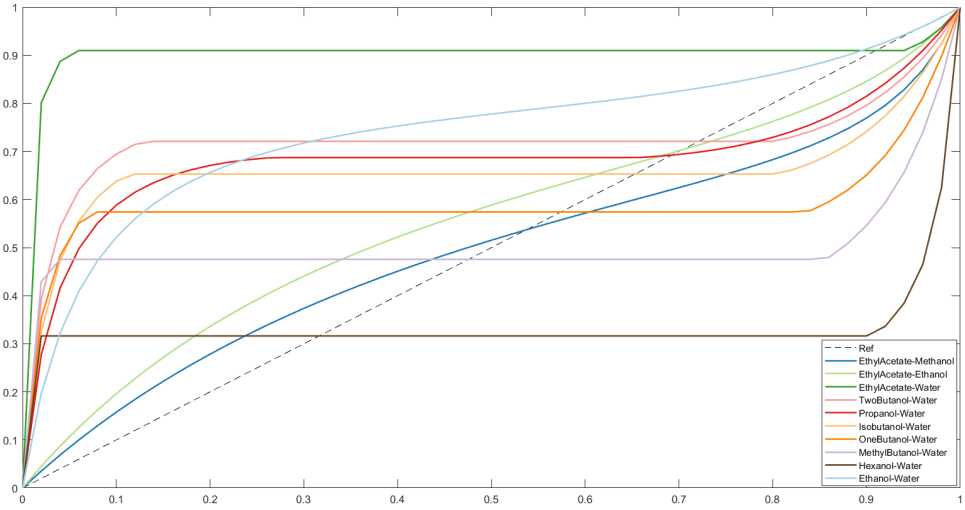

Figure 1: Plots of all possible azeotropes in mass basis

As experimental data will be available later in the thesis, it will be clear by looking at the mass fractions that only some of them will be relevant. The ethylacetate-methanol and ethylacetate-ethanol azeotropes might become important if the starting product contains a high quantity of ethylacetate, as it's a compound that can be found in the heads of distillation, and as such might be problematic when dealing with the distillate of the second column. On the other hand, the water-ethanol azeotrope is extremely relevant, as it prevents the complete separation of the two in the two columns, instead setting an upper limit of 95.6% ethanol (in mass).

1.4 Aim of the thesis

In this thesis, the distillation columns used at Acquavite S.p.A. for the production of grappa will be studied. The focus will be on the calculation of the efficiency of the two columns as well as their energy consumption, and some ideas in order to possibly improve the distillation train's efficiency and profitability will be provided.

Chapter 2 - Acquavite S.p.A.

2.1 The company

In 1996, Roberto Castagner established Acquavite S.p.A. in Visnà di Vazzola, located few kilometers away from Conegliano. In 1998, the decision to not focus on just selling bulk grappa was taken, starting to distribute their own products under the brand 'Castagner'. In the same year, the first barrels of grappa for long-term aging were set aside, being the first step to the construction of today's aging warehouse, where thousands of barrels are stored yearly.

The expansion of Acquavite S.p.A. in the retail industry lead to years of development to cater to an ever-increasing and diversified public. Starting from 2005, thanks to the partnership with many brands in the organized distribution and, in the last few years, to branching out towards the foreign markets, it was possible for the company to export their brand and products, often made to meet the needs of a specific niche in said market, in countries all around Europe, in Brasil, in the United States and Canada, and even in China and Korea.

In 2006, Roberto Castagner's idea of celebrating Italian figures who distinguished themselves in their respective field became reality. "Premio Fuoriclasse" is a yearly award conferred by a panel of 42 high-profile judges to the person who, according to them, enriched the society the most in their field of expertise. Among those who have been awarded can be found Piero Angela in 2020, Federica Pellegrini in 2018 and Andrea Bocelli in 2017.

Ever since 2014, no year has gone by without at least one award being received by Acquavite S.p.A. for their products. Ranging from awards such as "Miglior produttore di grappa" from the Wine Oscars, to "5 grappoli d'oro" from "Guida Bibenda Vini e Ristoranti d'Italia", to being awarded for the labels and packaging and even ranking highly in international competitions, Acquavite S.p.A.'s quality has been recognized on the international stage, so much so that it even made it within the top 100 spots of the *Forbes Italian Excellence* list.

2.2 Present production plant

While Acquavite S.p.A.'s focus is mainly on grappa, raw materials are also turned into secondary products, in order to cover for part of the raw materials and operation's costs and to reduce wastes. Figure 2 below briefly outlines the plant's production process from start to finish. Each block will be expanded upon afterwards.

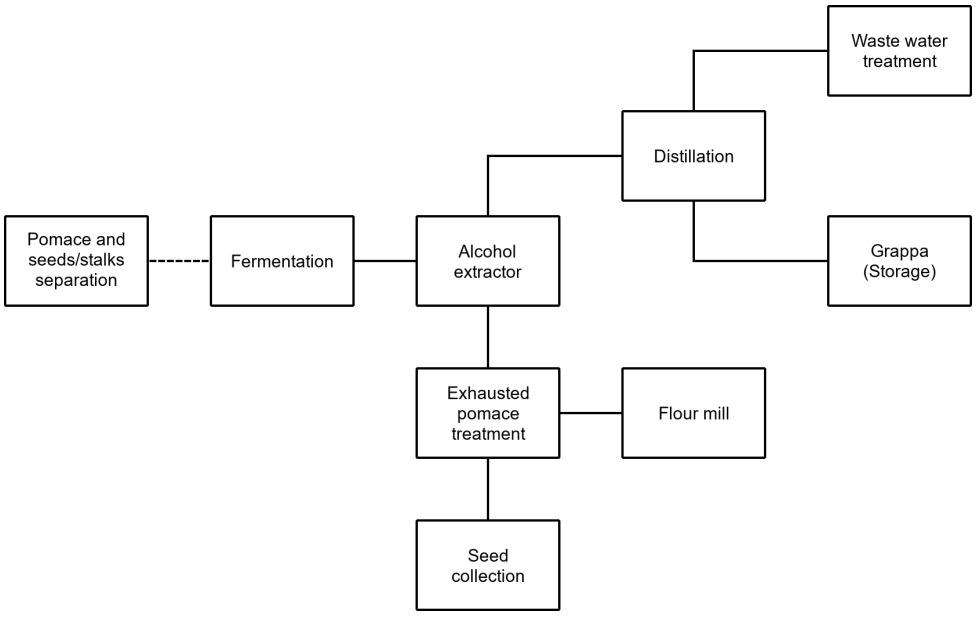

Figure 2: Block Flow Diagram of the plant at Acquavite S.p.A.

2.2.1 Separation of pomace and seeds and stalks

The first step consists in using mechanical agitators to separate pomace from grape seeds. This is not carried out for all the incoming raw materials, but only for white grapes' pomace. The reason can be traced back to how wine is made: for red wine, pomace is left within the wort for some days in order to extract the tannins, anthocyanins and polyphenols contained within the pomace. As this procedure is not carried out for white wines, white pomace is rich in these substances, especially polyphenols. These are widely known to have beneficial effect to the human body, especially on the skin [6], and are as such requested by cosmetics' manufacturers.

2.2.2 Fermentation

Fermentation is a very important step in grappa production process. The incoming pomace is first separated based on the variety, then it's inserted into food-grade plastic tubes thanks to a machine called *Grappasystem* that sprays a solution at basic pH over the pomace to reduce the amount of bacteria that would lead to sugars being turned into methanol. Instead, once the tube is full, selected yeast strands are introduced into it, before CO_2 is used to pressurize the tube in order to create an environment where pomace can be kept fresh for months and where alcohol fermentation can be controlled in order to produce as much ethanol and as little methanol as possible.

2.2.3 Alcohol recovery

Once pomace is ready for the next step, it's moved out of the tubes and into the alcohol extractor. Here, screw pumps move the pomace counter-current to steam in order to remove ethanol and the compounds that give grappa its scents and flavors, thus separating them from all the solids. The vapor-phase mixture, once cooled down to liquid phase, is known as phlegm. Its alcohol content varies between 15% and 30%, depending on the ethanol content in the raw material and the amount of steam used in the alcohol extractor. Being a liquid and being unable to ferment any further, phlegm is easier to conserve than pomace within steel containers, in case it isn't immediately needed for the following processes.

On the other hand, once pomace comes out from the other side of the alcohol extractor, very little to no alcohol is left inside it, while it's now rich in water, thus needing further treatment to further process it.

2.2.4 Exhausted pomace treatment - Seed collection and flour mill

As pomace comes out of the alcohol extractor, a press is used to squeeze it in batches to extract as much water as possible, water that will be treated in the water purifier that will be talked about below. The heaps of pomace move through a furnace, where it's completely dried. Grape seeds and dry skin are separated with the use of mechanical sieves in order to be processed: while the seeds are rich in oils and thus they will be stored away to be shipped to a facility to produce seed oil, the skin is sent to an in-house mill to be turned into flour.

Most of this flour will be sold as animal feed, if it meets the requirment to be recognized as animal feed. A part of it, however, will be used as fuel in the furnace to lower the need for outside sources of energy to dry the exhausted pomace. The amount of flour that can be used is under regulation, in order to keep below the limit of CO_2 emissions.

2.2.5 Distillation

Phlegm is the starting point for the production of grappa and, as explained earlier, distillation is needed in order to separate the *cuore* from the *teste* and the *code*. A train of two columns is needed to obtain such a result, and the specifications of the ones at Acquavite S.p.A. can be found in table 1 below, as well as a representation of the distillation train (Fig. 3).

Table 1: Columns data

	COL1	COL2
Number of Plates	48	58
Feed Plate	19	24
Diameter [m]	1	0.7
Temperature at the top [°C]	76	62.2
Temperature at the bottom [°C]	100.2	72.7
Feed temperature [°C]	44	45

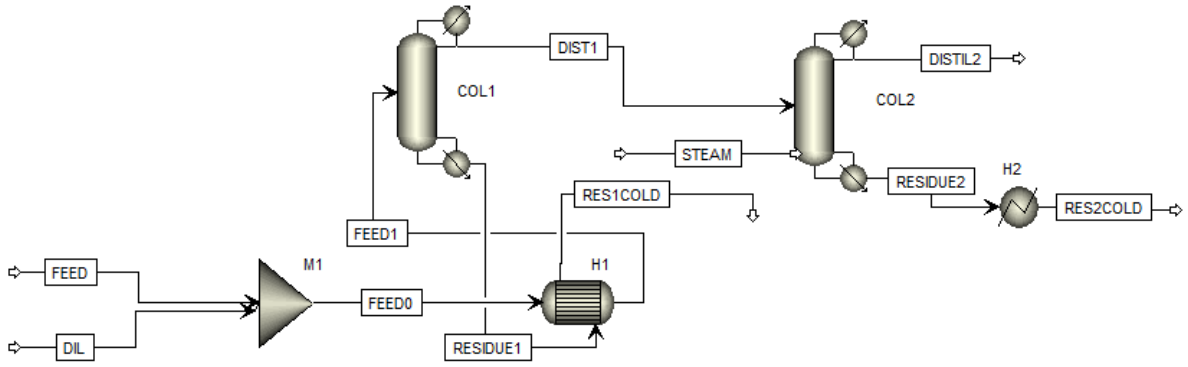

Figure 3: Representation of the present distillation train

Before entering in the first column, water is added to the phlegm in order to bring it to the desired alcohol content.

The first column is made of steel in the enrichment section, while the stripping section is made of copper, while the second column (demethylation column) is made entirely of copper. The residue stream of the first column is used to pre-heat the feed to 44°C from room temperature in order to recover some of the heat used within the column, before being sent to a water purifier. The distillate stream, on the other hand, is used as the feed for the second column after being fully condensed and brought down to 45°C, before being fed into the second column. Here, most of the methanol is removed from the top and, after total condensation, is sent to storage before being shipped away for further treatment, while the residue, containing mostly ethanol and water, is the desired product, that will be stored in steel drums until the aging process will start.

It's important to notice that the demethylation column's heating at the reboiler isn't provided just by the heat exchangers. Instead, direct steam is injected at the reboiler stage to both heat the mixture up and lower the alcohol content to the desired amount (around 80%), as it's less expensive than having a feed with an higher water content and heating it up at the reboiler and it would contain more of the higher-boiling impurities as well, making it a disadvantageous choice.

Figure 4: Picture of the distillation columns

2.2.6 Storage and aging

After grappa is cooled down, it's transferred into 100 hectoliter steel drums to finish. From here, when needed, it'll be taken out and moved to aging in barrels, or will undergo different treatments depending on the market's requests. In order to lower the alcohol content, osmotized water is added to reach the desired composition and from there, grappa is ready to be bottled up and sold.

2.2.7 Waste water treatment

After the heat exchanger to pre-heat the first column's feed, the residue from the first column has to be treated before being allowed back in the waters outside due to their high COD (Chemical Oxygen Demand) score. COD is defined as the amount of oxygen equivalents consumed in the chemical oxidation of organic matter by strong oxidant (e.g., potassium dichromate) [7], whose value in the residue stream ranges between 2000 and 3000, based on the raw materials' conditions.

Since the legal limit for water to be discharged in surface waters is a COD of 160 mg/L[8], it's necessary to proceed with treatment in order to decrease the amount of carbon-based compounds. Acquavite S.p.A. opted to use a conventional aerobic treatment plant that, thanks to selected strands of bacteria, converts the carbon-based and nitrogen-based compounds into products that are easier to precipitate with the use of flocculating agents. The sludge is collected (at a rate of around 8 m^3/h) before being shipped away to be turned into biofuel or compost.

Chapter 3 - Data collection and calculations

3.1 Instrumentation used

Data collection has been performed on a day during which the plant was working under a steady state regime. The samples taken for analysis were collected within minutes of each other. To find the density of the samples, a Gibertini hydrostatic balance (Error margin: $\pm 0.0015g$) was used, while for composition, the instrument used was a Clarus 580 Gaschromatograph (Perkin Elmer) (Error margin: $\pm 6\%$).

For the instrumentation within the column, temperature data were obtained via Seneca PT100 temperature probes (Operating range: 0-120°C. Error margin: $\pm 0.2°C$), alcohol contents were given by Bopp & Reuther Messtechnik Density meters Series 1.3. (Operating range: -40-150 °C. Error margin: $\pm 0.1° Kg/m^3$) while the volumetric flow rates came from ROTA-YOKAGAWA RAMC02-D4SS flow meters (Error margin: $\pm 2.5\%$).

Volumetric flow rates were taken after the condensers, while density was measured at 20°C.

3.2 Collected data

The collected data for temperatures, densities, volumetric flow rates and composition are available in the tables below.

Table 2: Collected temperature data [°C]

Feed (before heat exchanger)	25
Feed (after heat exchanger)	44
Dist1	45
Res1	100.2
Dist2	45
Res2	62.2

Table 3: Collected density data [kg/dm^3]

Feed (after diluition)	0.9732
Dist1	0.8164
Dist2	0.8608
Res2	0.8620

Table 4: Collected volumetric flow rates data [l/h]

Feed (pre-diluition)	1300
Feed (after diluition)	1500
Dist1	340
Dist2	11
Res2	390
Reflux1	1660
Reflux2	639

Table 5: Collected composition data [mg/100mlEtOH]

	Feed (after diluition)	Dist1	Res2
Acetaldehyde	67.77	54.79	0.08
Ethyl-Acetate	520.70	551.81	8.32
2-Butanol	66.47	73.68	95.68
Propanol	76.84	64.27	87.00
Isobutanol	82.91	26.49	37.88
1-Butanol	2.73	6.12	0.02
Methyl-butanol	177.24	1.07	0.01
Ethyl-Lactate	11.69	0.30	0.20
Hexanol	6.18	0.00	0.00
Methanol	1403.03	1419.70	278.29

3.3 Conversions to mass flow rates

When water and ethanol are mixed, the volume of the mixture differs from the sum of the two single volumes. As such, it's impossible to use volume flow rates for the mass balances, making it necessary to convert all flow rates into mass flow rates. The process requires solving a system of equations and will be explained below.
Firstly, the volume flow rates have to be converted to mass flow rates with the following formula:

$$\dot{V} = \dot{W} \cdot \rho \tag{1}$$

Having the densities and the mass flow rates, it's possible to set up a system of equation for each of the three streams in order to find the mass flow rates of each compound.
The first equation is a very simple one:

$$\sum_k \dot{W}_k = \dot{W}_{Tot} \tag{2}$$

with k being every component in the mixture. This can further be manipulated to express the dependence of the compounds from the ethanol flow rate:

$$\dot{W}_{Et} + \dot{W}_W + \frac{\dot{W}_{Et}}{78.9 \cdot 1000} \cdot (t_{Ac} + t_{EA} + t_{2B} + t_{Pr} + t_{IB} + t_{1B} + t_{MB} + t_{EL} + t_{Hex} + t_{Me}) = \dot{W}_{Tot} \tag{3}$$

where t is the concentration value obtained from the gas chromatograph and available in table 5.
The second equation relates the mixture's excess molar volume to the molar fraction of ethanol in the ethanol/water mixture. Since the amount of other compounds in the analyzed streams is low enough, the excess volume of mixture between all of them can be ignored in first approximation.

The definition of excess volume of a mixture is as follows[9]:

$$V^E = \sum_i [(\frac{1}{\rho_{Mix}} - \frac{1}{\rho_i}) \cdot x_i \cdot MW_i] \tag{4}$$

that, for a binary mixture of water and ethanol, like in this case, can be rewritten as:

$$V^E = [(\frac{1}{\rho_{Mix}} - \frac{1}{\rho_W}) \cdot (1 - x_{Et}) \cdot MW_W] + [(\frac{1}{\rho_{Mix}} - \frac{1}{\rho_{Et}}) \cdot x_{Et} \cdot MW_{Et}] \tag{5}$$

with n being the molar fraction of ethanol within the mixture, related to the mass fraction by the following equation:

$$n = \frac{X_{Et}}{X_{Et} + X_W} = \frac{\dot{W}_{Et}/MW_{Et}}{\dot{W}_{Et}/MW_{Et} + \dot{W}_W/MW_W} \tag{6}$$

In literature[9], experimental data correlating the excess molar volume to the mole ratio of ethanol and water mixtures are available. Fitting those data, it's possible to obtain an analytical correlation to extimate the excess molar volume based on the mole ratio between the compounds. The fitting in figure 5 was done using MatLab's curve fitting tool, obtaining equation 7, that almost perfectly fits the experimental data in the interval of interest [0;1].

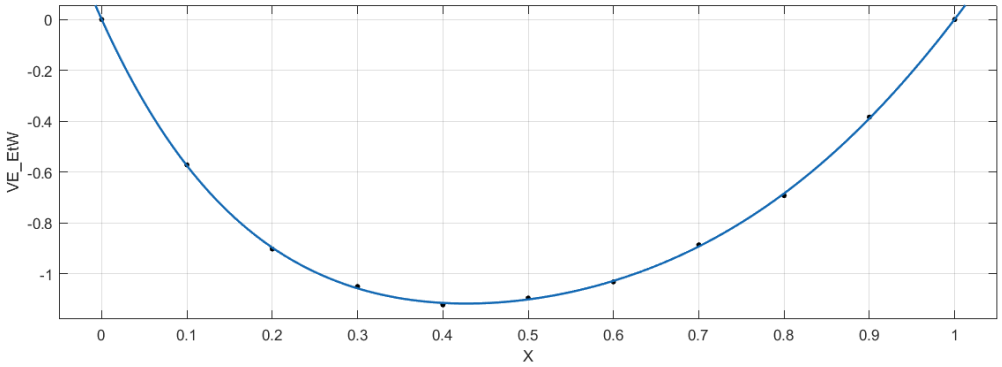

Figure 5: Fitting of excess molar volume data

$$V^E = -5.139 \cdot x_{Et}^5 + 18.84 \cdot x_{Et}^4 - 25.31 \cdot x_{Et}^3 + 19.03 \cdot x_{Et}^2 - 7.428 \cdot x_{Et} + 0.0006435 \tag{7}$$

Solving the system composed of equation 3, equation 5, equation 6 and equation 7, the values of $\dot{W}$ and $\dot{E}t$ can be found. From there, the values of the mass flow rates for the other compounds can be easily found using equation 8:

$$\dot{W}_k = \frac{\dot{W_{Et}}}{78.9 \cdot 1000} \cdot t_k \tag{8}$$

The results are available in table 6.

Table 6: Partial mass flow rates in [kg/h]

	Feed (after diluition)	Dist1	Res2
Acetaldehyde	0.21	0.17	0.00
Ethyl-Acetate	1.65	1.71	0.03
2-Butanol	0.21	0.23	0.29
Propanol	0.24	0.20	0.27
Isobutanol	0.26	0.08	0.11
1-Butanol	0.01	0.02	0.00
Methyl-butanol	0.56	0.00	0.00
Ethyl-Lactate	0.04	0.00	0.00
Hexanol	0.02	0.00	0.00
Methanol	4.43	4.41	0.88
Ethanol	249.50	245.40	242.87
Water	1208.00	25.23	91.72

3.4 Mass balances

In order to perform calculations on the efficiency of the two columns, it's necessary to obtain the mass flow rates of the remaining streams. This can easily be done by writing the mass balances relative to the colums, as shown below.

For the first column:

$$\begin{aligned}
\dot{W}_{Ac,R1} &= \dot{W}_{Ac,F} - \dot{W}_{Ac,D1} \\
\dot{W}_{EA,R1} &= \dot{W}_{EA,F} - \dot{W}_{EA,D1} \\
\dot{W}_{2B,R1} &= \dot{W}_{2B,F} - \dot{W}_{2B,D1} \\
\dot{W}_{Pr,R1} &= \dot{W}_{Pr,F} - \dot{W}_{Pr,D1} \\
\dot{W}_{IB,R1} &= \dot{W}_{IB,F} - \dot{W}_{IB,D1} \\
\dot{W}_{1B,R1} &= \dot{W}_{1B,F} - \dot{W}_{1B,D1} \\
\dot{W}_{MB,R1} &= \dot{W}_{MB,F} - \dot{W}_{MB,D1} \\
\dot{W}_{EL,R1} &= \dot{W}_{EL,F} - \dot{W}_{EL,D1} \\
\dot{W}_{Hex,R1} &= \dot{W}_{Hex,F} - \dot{W}_{Hex,D1} \\
\dot{W}_{Met,R1} &= \dot{W}_{Met,F} - \dot{W}_{Met,D1} \\
\dot{W}_{Et,R1} &= \dot{W}_{Et,F} - \dot{W}_{Et,D1} \\
\dot{W}_{W,R1} &= \dot{W}_{W,F} - \dot{W}_{W,D1}
\end{aligned} \tag{9}$$

For the second column:

$$
\begin{aligned}
\dot{W}_{Ac,D2} &= \dot{W}_{Ac,D1} - \dot{W}_{Ac,R2} \\
\dot{W}_{EA,D2} &= \dot{W}_{EA,D1} - \dot{W}_{EA,R2} \\
\dot{W}_{2B,D2} &= \dot{W}_{2B,D1} - \dot{W}_{2B,R2} \\
\dot{W}_{Pr,D2} &= \dot{W}_{Pr,D1} - \dot{W}_{Pr,R2} \\
\dot{W}_{IB,D2} &= \dot{W}_{IB,D1} - \dot{W}_{IB,R2} \\
\dot{W}_{1B,D2} &= \dot{W}_{1B,D1} - \dot{W}_{1B,R2} \\
\dot{W}_{MB,D2} &= \dot{W}_{MB,D1} - \dot{W}_{MB,R2} \\
\dot{W}_{EL,D2} &= \dot{W}_{EL,D1} - \dot{W}_{EL,R2} \\
\dot{W}_{Hex,D2} &= \dot{W}_{Hex,D1} - \dot{W}_{Hex,R2} \\
\dot{W}_{Met,D2} &= \dot{W}_{Met,D1} - \dot{W}_{Met,R2} \\
\dot{W}_{Et,D2} &= \dot{W}_{Et,D1} - \dot{W}_{Et,R2} \\
\dot{W}_{W,D2} &= \dot{W}_{Tot,D2} - \sum_{K} \dot{W}_{K,D2} \\
\dot{W}_{W,S} &= \dot{W}_{W,D2} + \dot{W}_{W,R2} - \dot{W}_{W,D1}
\end{aligned}
\tag{10}
$$

The calculated mass flow rates of all streams are reported in table 7 and are converted in molar flow rates in table 8. In addition, table 9 shows the mass fractions of each component within each stream. Negative values can be attributed to the systematic error caused by the measurement devices' confidence intervals due to their small values.

Table 7: Mass flow rates in [kg/h]

	Feed (after diluition)	Dist1	Res1	Dist2	Res2	S
Acetaldehyde	0.21	0.17	0.04	0.17	0.00	0.00
Ethyl-Acetate	1.65	1.71	-0.07	1.69	0.03	0.00
2-Butanol	0.21	0.23	-0.02	-0.07	0.29	0.00
Propanol	0.24	0.20	0.04	-0.07	0.27	0.00
Isobutanol	0.26	0.08	0.18	-0.03	0.11	0.00
1-Butanol	0.01	0.02	-0.01	0.02	0.00	0.00
Methyl-butanol	0.56	0.00	0.56	0.00	0.00	0.00
Ethyl-Lactate	0.04	0.00	0.04	0.00	0.00	0.00
Hexanol	0.02	0.00	0.02	0.00	0.00	0.00
Methanol	4.43	4.41	0.02	3.53	0.88	0.00
Ethanol	249.50	245.40	4.00	2.62	242.87	0.00
Water	1208.00	25.23	1182.73	1.60	91.72	68.08
Total	1465.2	277.58	1187.53	9.47	336.18	68.8

Table 8: Molar flow rates in [kmol/h]

	Feed (after diluition)	Dist1	Res1	Dist2	Res2	S
Acetaldehyde	0.000	0.000	0.000	0.000	0.000	0.000
Ethyl-Acetate	0.002	0.002	0.000	0.002	0.000	0.000
2-Butanol	0.000	0.000	0.000	0.000	0.000	0.000
Propanol	0.000	0.000	0.000	0.000	0.000	0.000
Isobutanol	0.000	0.000	0.000	0.000	0.000	0.000
1-Butanol	0.000	0.000	0.000	0.000	0.000	0.000
Methyl-butanol	0.001	0.000	0.001	0.000	0.000	0.000
Ethyl-Lactate	0.000	0.000	0.000	0.000	0.000	0.000
Hexanol	0.000	0.000	0.000	0.000	0.000	0.000
Methanol	0.138	0.138	0.000	0.110	0.028	0.000
Ethanol	5.416	5.329	0.087	0.057	5.272	0.000
Water	67.057	1.401	65.656	0.089	5.091	3.779
Total	72.614	6.870	65.744	0.258	10.391	3.779

Table 9: Mass fractions

	Feed (after diluition)	Dist1	Res1	Dist2	Res2	S
Acetaldehyde	0.000	0.000	0.000	0.018	0.000	0.000
Ethyl-Acetate	0.001	0.006	0.000	0.178	0.000	0.000
2-Butanol	0.000	0.000	0.000	-0.007	0.000	0.000
Propanol	0.000	0.000	0.000	-0.007	0.000	0.000
Isobutanol	0.000	0.000	0.000	-0.004	0.000	0.000
1-Butanol	0.000	0.000	0.000	0.002	0.000	0.000
Methyl-butanol	0.000	0.000	0.000	0.000	0.000	0.000
Ethyl-Lactate	0.000	0.000	0.000	0.000	0.000	0.000
Hexanol	0.000	0.000	0.000	0.000	0.000	0.000
Methanol	0.003	0.016	0.000	0.373	0.003	0.000
Ethanol	0.170	0.885	0.003	0.277	0.723	0.000
Water	0.825	0.091	0.996	0.169	0.273	0.000

Chapter 4 - Efficiency calculations of the distillation train

In order to calculate the efficiency of the column, McCabe-Thiele's graphical method will be used, applying it to the system of interest for the column: water-ethanol for the first column and ethanol-methanol for the second column. AspenPlus has been used to provide for the equilibrium curves for both systems at 1 atm.

4.1 First column

It's necessary to calculate the mass fraction of ethanol in the three streams in exam (x_F, x_D and x_R) in order to proceed. These can be easily calculated:

$$x_F = \frac{\dot{W}_{F,Et}}{\dot{W}_{F,Et} + \dot{W}_{F,W}} = \frac{249.50\frac{kg}{h}}{249.50\frac{kg}{h} + 1208\frac{kg}{h}} = 0.171$$

$$x_D = \frac{\dot{W}_{D1,Et}}{\dot{W}_{D1,Et} + \dot{W}_{D1,W}} = \frac{245.40\frac{kg}{h}}{245.40\frac{kg}{h} + 25.23\frac{kg}{h}} = 0.907 \tag{11}$$

$$x_R = \frac{\dot{W}_{R1,Et}}{\dot{W}_{R1,Et} + \dot{W}_{R1,W}} = \frac{4.00\frac{kg}{h}}{4.00\frac{kg}{h} + 1182.73\frac{kg}{h}} = 0.003$$

Another necessary information in order to proceed is the mixture's boiling temperature. This has been calculated using Antoine's correlation, setting the saturated vapor pressure to 1 atm (760 mmHg) thus finding the boiling temperature as a result, as shown in equation 12, where i is the i-th component in the feed, X its mole fraction, A, B and C its Antoine coefficients and T the boiling temperature of the feed:

$$log_{10}(760) = \sum_i [\gamma_i \cdot X_i \cdot (A_i - \frac{B_i}{T + C_i})] \tag{12}$$

For the Feed stream, solving this equation returns a value of temperature equal to 97.53 °C. Likewise, the heat capacity of the mixture has to be calculated as a weighted average of each component's heat capacity relative to their molar fraction. The same procedure has to performed with the mixture's vaporization enthalpy, as seen in equations 13.

$$\begin{aligned} c_{P,Mix} &= \sum_i c_{P,i} \cdot X_i \\ \lambda_{Mix} &= \sum_i \lambda_i \cdot X_i \end{aligned} \tag{13}$$

For the Feed stream, $c_P = 7.85 \cdot 10^4 \frac{J}{kmol \cdot K}$ while $\lambda = 4.05 \cdot 10^4 \frac{kJ}{kmol}$. McCabe-Thiele's construction requires the calculation of the feed stream's vapor fraction φ_F as

$$\varphi_F = -\frac{c_{P,Feed}}{\lambda_{Feed}} \cdot (T_{boil,Feed} - T_{Feed}) = -0.104 \tag{14}$$

As expected, the value is between 0 and -1, since it's a liquid below its boiling temperature.

Once the points F(x_F,x_F), D(x_D,x_D) and R(x_R,x_R) are found on the plot, the construction lines can be built. The feed line is the line passing through point F and whose slope can be calculated with equation 15

$$m_{F,Col1} = -\frac{1-\varphi_F}{\varphi_F} = 10.615 \tag{15}$$

The enrichment line passes through point D, and its slope is given by equation 16

$$m_E = \frac{r}{r+1} \tag{16}$$

With r being the reflux ratio of the column, defined as the ratio between the flow rate of the stream going back into the column and the flow rate of the distillate stream. Since the condensers are total condensers, the density of the two streams is the same, so it's indifferent whether mass flow rates or volume flow rates are used, in this case, since the ratio r will be the same. As such, equation 16 can be written as

$$m_{E,Col1} = \frac{\dot{V}_{Ref1}/\dot{V}_{D1}}{(\dot{V}_{Ref1}/\dot{V}_{D1})+1} = \frac{1600/340}{(1600/340)+1} = \frac{4.882}{4.882+1} = 0.83 \tag{17}$$

The stripping line is then drawn as the line passing through both the point S and the point where the enrichment and feed line meet. The rest of the construction involves horizontal lines between the operating line and the equilibrium line, starting from point D and reaching point S, can be found in figures 6 and 7.

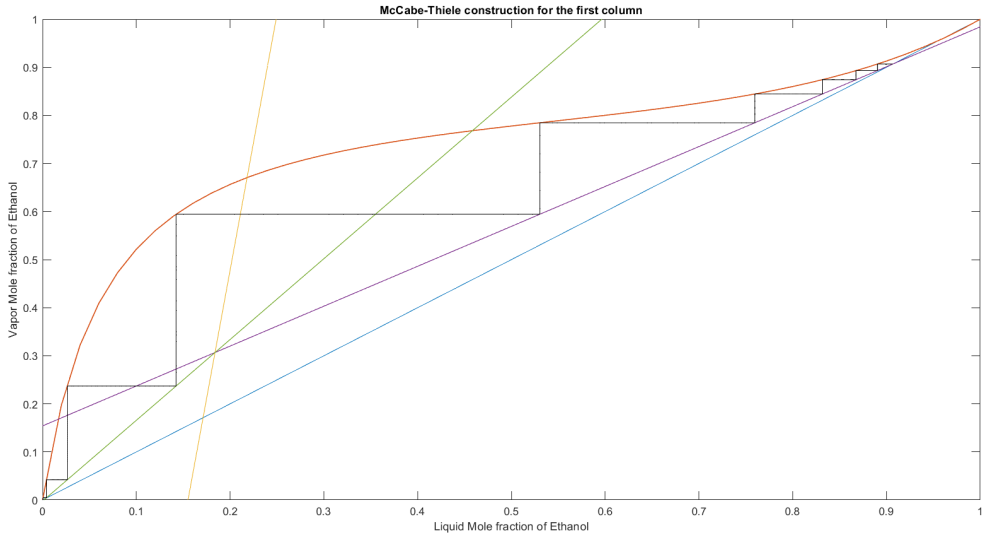

Figure 6: McCabe-Thiele construction for Column 1

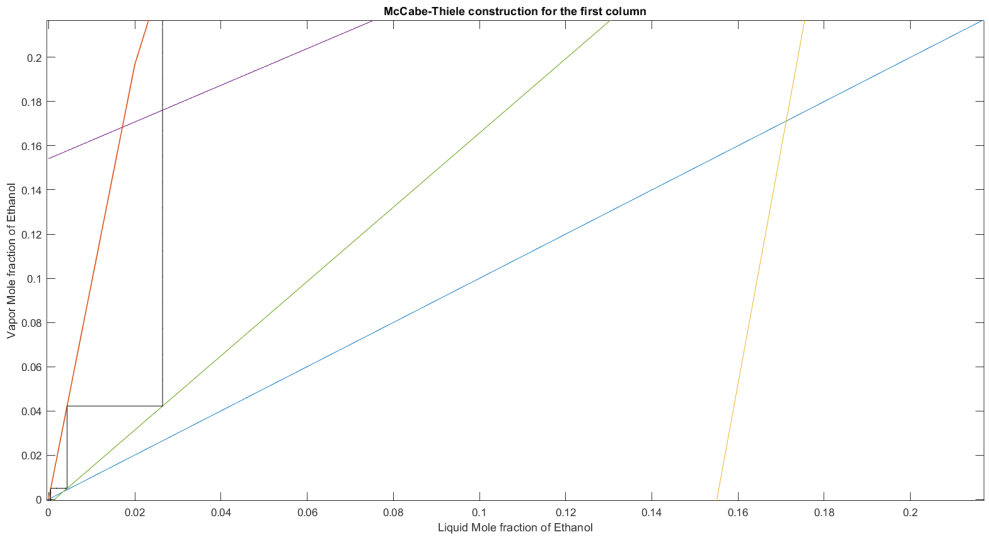

Figure 7: Zoom of McCabe-Thiele construction for Column 1 at the end of the stripping section

The number of times the construction lines touch the equilibrium line is 9, which is also the number of ideal trays, according the McCabe-Thiele's method. The column's efficiency compared to the ideal case can be calculated as the ratio between the number of ideal trays and the number of real trays. In this case:

$$\eta_{Col1} = \frac{\#\text{ of ideal trays}}{\#\text{ of real trays}} = \frac{9}{48} = 0.1875 \tag{18}$$

The calculated efficiency of the first column is 18.75%. This is a low value of efficiency, meaning that the same ethanol-water separation can be achieved with a smaller column. It should be noted, however, that the column's efficiency is very sensitive to the mass fraction of ethanol in the distillate, as the closer to the azeotrope the mixture is, the closer the equilibrium curve is to the operating curve and the more trays are needed to reach that degree of separation. In addition, water and ethanol are not the only compounds in the mixture. In particular, compounds like 2-Butanol, Propanol and Isobutanol provide necessary organoleptic properties to the final product, making it optimal to use an higher number of plates to separate those compounds from water as well.

4.2 Second column

The same process can be done for the second column. However, the system in exam is not water-ethanol but instead ethanol-methanol. As such, the equilibrium curve changes to that of the new system.

$$x_F = \frac{\dot{W}_{D1,Met}}{\dot{W}_{D1,Met} + \dot{W}_{D1,Et}} = \frac{4.41\frac{kg}{h}}{245.40\frac{kg}{h} + 4.41\frac{kg}{h}} = 0.018$$

$$x_D = \frac{\dot{W}_{D2,Met}}{\dot{W}_{D2,Met} + \dot{W}_{D2,Et}} = \frac{3.53\frac{kg}{h}}{3.53\frac{kg}{h} + 2.62\frac{kg}{h}} = 0.574 \tag{19}$$

$$x_R = \frac{\dot{W}_{R2,Met}}{\dot{W}_{D2,Met} + \dot{W}_{R2,Et}} = \frac{0.88\frac{kg}{h}}{0.88\frac{kg}{h} + 242.87\frac{kg}{h}} = 0.004$$

$$\begin{aligned} c_{P,D1} &= 1.09 \cdot 10^5 \frac{J}{kmol \cdot K} \\ \lambda_{D1} &= 3.92 \cdot 10^4 \frac{kJ}{kmol} \\ T_{Boil,D1} &= 81.16°C \\ \varphi_F &= -\frac{c_{P,D1}}{\lambda_{D1}} \cdot (T_{boil,D1} - T_{D1}) = -0.100 \end{aligned} \tag{20}$$

$$\begin{aligned} m_{F,Col1} &= -\frac{1-\varphi_F}{\varphi_F} = 10.98 \\ m_{E,Col1} &= \frac{\dot{V}_{Ref2}/\dot{V}_{D2}}{(\dot{V}_{Ref2}/\dot{V}_{D2}) + 1} = \frac{639/11}{(639/11 + 1} = \frac{58.09}{58.09 + 1} = 0.98 \end{aligned} \tag{21}$$

The resulting construction plots are available in figures 8 and 9.

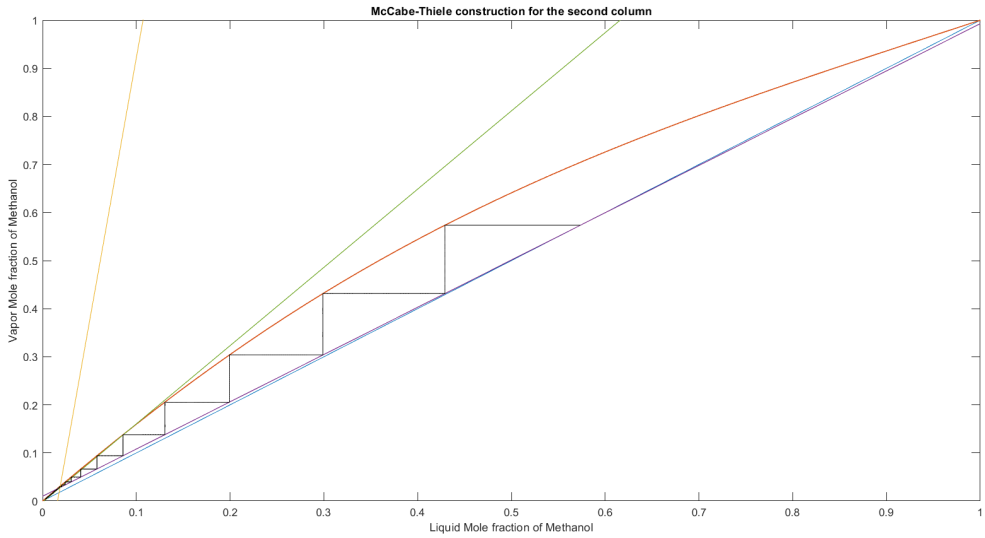

Figure 8: McCabe-Thiele construction for Column 2

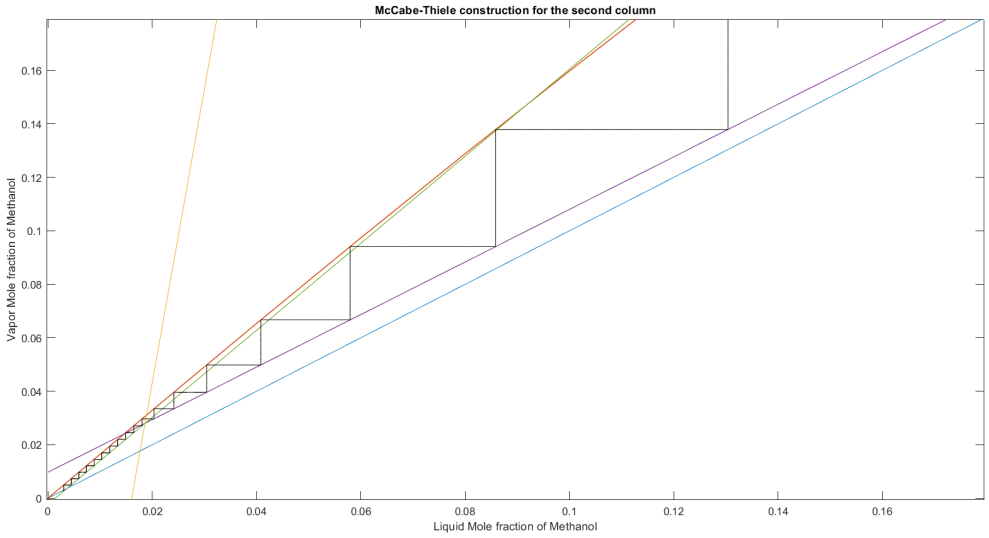

Figure 9: Zoom of McCabe-Thiele construction for Column 2 at the end of the stripping section

The number of ideal trays is 21. The efficiency of the column can be calculated with the same formula used earlier:

$$\eta_{Col2} = \frac{\#\text{ of ideal trays}}{\#\text{ of real trays}} = \frac{21}{58} = 0.3621 \tag{22}$$

That translates to a 36.21% efficiency. While it may seem a low efficiency, it is necessary for the demethylation column to be able to separate most of the methanol within the Dist1 stream in order to comply with Italian laws. In addition, methanol and ethanol amount is never fixed due to the impossibility to perfectly control the pomace fermentation, that might at times produce more methanol than the case in exam. Lastly, different countries have different laws in place regarding the allowed amount of methanol in alcoholic drinks [10]. Lower allowed methanol contents would mean an higher degree of separation and thus more trays needed to perform it.

4.3 Usage of utilities within the distillation train

As seen in figure 3, the plant's heat transfer happens through two heat exchangers, and the two columns' reboilers and condensers. The first heat exchanger is of little interest for the calculations of utilities, as it involves the feed stream and the residue1 stream, and as such will not be considered in this thesis. The second heat exchanger is necessary to cool down the final product (grappa) before storage. The two columns' condensers are total condensers, using cold water to bring the reflux streams to liquid state. The first column's heating is provided by a coil in the reboiler stage, while the second column is heated by both a coil at the reboiler stage and steam entering at the same stage. Table 10 below contains the properties of the available utilities.

Table 10: Utilities' properties

Cold water temperature [°C]	15.3
Saturated vapor temperature [°C]	140
Saturated vapor pressure [bar]	3.61

For the heat exchanger:

Table 11: Temperatures for the heat exchanger on Residue2 stream

	Temperature [°C]
Utilities inlet	15.3
Utilities outlet	19
Grappa inlet	62.2
Grappa outlet	20

$$\begin{aligned} Q_{HeatEX} &= (c_{P,W} \cdot \dot{W}_{W,D2} + c_{P,Et} \cdot \dot{W}_{Et,D2} + c_{P,Met} \cdot \dot{W}_{Met,D2}) \cdot (T_{G,Out} - T_{G,In}) = \\ &= (4186 \frac{J}{Kg \cdot K} \cdot 91.72 \frac{Kg}{h} + 2438 \frac{J}{Kg \cdot K} \cdot 242.87 \frac{Kg}{h} + 2512 \frac{J}{Kg \cdot K} \cdot 0.88 \frac{Kg}{h}) \cdot \\ &\cdot (62.2°C - 20°C) = \\ &= -4.13 \cdot 10^7 \frac{J}{h} = -11.5kW \end{aligned} \tag{23}$$

For the first column's condenser:

Table 12: Temperatures for the first column's condenser

	Temperature [°C]
Utilities inlet	15.3
Utilities outlet	65
Condenser inlet	76
Condenser outlet	45

$$\begin{aligned} Q_{C1} = &(c_{P,W} \cdot \dot{W}_W + c_{P,Et} \cdot \dot{W}_{Et} + c_{P,Met} \cdot \dot{W}_{Met}) \cdot (T_{C,Out} - T_{C,In}) + \\ &+ (C_{V,W} \cdot \dot{W}_W + C_{V,Et} \cdot \dot{W}_{Et} + C_{V,Met} \cdot \dot{W}_{Met}) \end{aligned} \tag{24}$$

Assuming the density and composition of the reflux and the distillate are identical, equation 24 can be rewritten as the sum of two distinct contributes, one coming from the condensation of the vapor phase and the other coming from the cooling. The two parts of the equation will be split for ease of manageability:

$$\begin{aligned} Q_{C1,Cool} = &(c_{P,W} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{W,D1} + \\ &+ c_{P,Et} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{Et,D1} + \\ &+ c_{P,Met} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{Met,D1} + \\ &+ c_{P,EA} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{EA,D1}) \cdot (T_{C,Out} - T_{C,In}) = \\ &= (4186 \frac{J}{kg \cdot K} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.091 + \\ &+ 2438 \frac{J}{kg \cdot K} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.885 + \\ &+ 2512 \frac{J}{kg \cdot K} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.016 + \\ &+ 1904 \frac{J}{kg \cdot K} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.006) \cdot (45 - 76)°C = \\ &= -1.36 \cdot 10^8 \frac{J}{h} = -37.8 kW \end{aligned} \tag{25}$$

$$\begin{aligned} Q_{C1,Cond} = &(-c_{V,W} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{W,D1} - \\ &- c_{V,Et} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{Et,D1} + \\ &- c_{V,Met} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{Met,D1}) + \\ &- c_{V,EA} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{EA,D1}) = \\ &= (-2257 \frac{kJ}{kg} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.091 + \\ &- 855 \frac{kJ}{kg} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.885 + \\ &- 1098.3 \frac{kJ}{kg} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.016) + \\ &- 404 \frac{kJ}{kg} \cdot (1660 + 340) \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.006) = \\ &= -1.60 \cdot 10^6 \frac{kJ}{h} = -1.60 \cdot 10^9 \frac{J}{h} = -444.4 kW \end{aligned} \tag{26}$$

Thus, adding the two contributes up, the result is:

$$Q_{C1,Tot} = Q_{C1,Cool} + Q_{C1,Cond} = -37.8kW - 444.4kW = -482.2kW = -1.74 \cdot 10^9 \frac{J}{h} \quad (27)$$

Where the negative sign indicates that it's energy that has to be taken away from the system by cooling it.

The same procedure can be performed for the second column's condenser:

Table 13: Temperatures for the second column's condenser

	Temperature [°C]
Utilities inlet	15.3
Utilities outlet	52.9
Condenser inlet	62.2
Condenser outlet	45

$$\begin{aligned} Q_{C2} = &(c_{P,W} \cdot \dot{W}_W + c_{P,Et} \cdot \dot{W}_{Et} + c_{P,Met} \cdot \dot{W}_{Met} + c_{P,EA} \cdot \dot{W}_{EA}) \cdot (T_{C,Out} - T_{C,In}) \\ &- (C_{V,W} \cdot \dot{W}_W + C_{V,Et} \cdot \dot{W}_{Et} + C_{V,Met} \cdot \dot{W}_{Met} + C_{V,EA} \cdot \dot{W}_{EA}) \end{aligned} \quad (28)$$

With the same assumption as the first condenser's case, the following can be written:

$$\begin{aligned} Q_{C2,Cool} = &(c_{P,W} \cdot (\dot{V}_{Ref2} + \dot{V}_{D2}) \cdot \rho_{D2} \cdot x_{W,D2} + \\ &+ c_{P,Et} \cdot (\dot{V}_{Ref2} + \dot{V}_{D2}) \cdot \rho_{D2} \cdot x_{Et,D2} + \\ &+ c_{P,Met} \cdot (\dot{V}_{Ref2} + \dot{V}_{D2}) \cdot \rho_{D2} \cdot x_{Met,D2} + \\ &+ c_{P,EA} \cdot (\dot{V}_{Ref2} + \dot{V}_{D2}) \cdot \rho_{D2} \cdot x_{EA,D2}) \cdot (T_{C,Out} - T_{C,In}) = \\ &= (4186 \frac{J}{kg \cdot K} \cdot (650) \frac{dm^3}{h} \cdot 0.8608 \frac{kg}{dm^3} \cdot 0.169 + \\ &+ 2438 \frac{J}{kg \cdot K} \cdot (650) \frac{dm^3}{h} \cdot 0.8608 \frac{kg}{dm^3} \cdot 0.277 + \\ &+ 2512 \frac{J}{kg \cdot K} \cdot (650) \frac{dm^3}{h} \cdot 0.8608 \frac{kg}{dm^3} \cdot 0.373 + \\ &+ 1904 \frac{J}{kg \cdot K} \cdot (650) \frac{dm^3}{h} \cdot 0.8608 \frac{kg}{dm^3} \cdot 0.178) \cdot \\ &\cdot (45 - 62.2)°C = \\ &= -2.56 \cdot 10^7 \frac{J}{h} = -7.11kW \end{aligned} \quad (29)$$

$$
\begin{aligned}
Q_{C2,Cond} = (&-c_{V,W} \cdot (\dot{V}_{Ref2} + \dot{V}_{D2}) \cdot \rho_{D2} \cdot x_{W,D2} - \\
&- c_{V,Et} \cdot (\dot{V}_{Ref2} + \dot{V}_{D2}) \cdot \rho_{D2} \cdot x_{Et,D2} + \\
&- c_{V,Met} \cdot (\dot{V}_{Ref2} + \dot{V}_{D2}) \cdot \rho_{D2} \cdot x_{Met,D2} +) = \\
&- c_{V,EA} \cdot (\dot{V}_{Ref2} + \dot{V}_{EA}) \cdot \rho_{D2} \cdot x_{EA,D2} = \\
&= (-2257\frac{kJ}{kg} \cdot (650)\frac{dm^3}{h} \cdot 0.8608\frac{dm^3}{h} \cdot 0.169 - \\
&- 855\frac{kJ}{kg} \cdot (650)\frac{dm^3}{h} \cdot 0.8608\frac{dm^3}{h} \cdot 0.277 - \\
&- 1098.3\frac{kJ}{kg} \cdot (650)\frac{dm^3}{h} \cdot 0.8608\frac{dm^3}{h} \cdot 0.273 - \\
&- 404\frac{kJ}{kg} \cdot (650)\frac{dm^3}{h} \cdot 0.8608\frac{dm^3}{h} \cdot 0.178) = \\
&= -5.54 \cdot 10^5 \frac{kJ}{h} = -5.54 \cdot 10^8 \frac{J}{h} = -153.89kW
\end{aligned}
\tag{30}
$$

And adding the two contributes:

$$
Q_{C2,Tot} = Q_{C2,Cool} + Q_{C2,Cond} = -7.11kW - 153.89kW = -161kW = -5.80 \cdot 10^8 \frac{J}{h} \tag{31}
$$

The calculation of the energy needed at the reboiler is done with equation 32:

$$
Q_{Reb} = -Q_C - \lambda \cdot \varphi \cdot \dot{X}_{Tot} \tag{32}
$$

For the first column, this reduces to:

$$
\begin{aligned}
Q_{Reb1} &= -Q_{C1} - \lambda_2 \cdot \varphi_2 \cdot \dot{X}_{D1} = \\
&= 1.74 \cdot 10^9 \frac{J}{h} - 3.92 \cdot 10^7 \frac{J}{kmol} \cdot (-0.104) \cdot 72.614\frac{kmol}{h} = \\
&= 2.05 \cdot 10^9 \frac{J}{h} = 569.44kW
\end{aligned}
\tag{33}
$$

Likewise, for the second column, equation 32 becomes:

$$
\begin{aligned}
Q_{Reb2} &= -Q_{C2} - \lambda_2 \cdot \varphi_2 \cdot \dot{X}_{D1} = \\
&= 5.80 \cdot 10^8 \frac{J}{h} - 4.05 \cdot 10^7 \frac{J}{kmol} \cdot (-0.100) \cdot 6.870\frac{kmol}{h} = \\
&= 6.08 \cdot 10^8 \frac{J}{h} = 188.89kW
\end{aligned}
\tag{34}
$$

However, that doesn't take into account the enrgy provided by the steam. As such, it will be calculated separately and added to the result of equation 34.

$$
\begin{aligned}
Q_{DirectSteam} &= \dot{W}_W \cdot c_{P,Steam} \cdot (T_{In} - T_{Boil}) + \dot{W}_W \cdot c_{V,W} + \dot{W}_W \cdot c_{P,W} \cdot (T_{Boil} - T_{Out}) = \\
&= \dot{W}_{W,S} \cdot (c_{P,Steam} \cdot (T_{In} - T_{Boil}) + \cdot c_{V,W} + c_{P,W} \cdot (T_{Boil} - T_{Out}) = \\
&= 68.8\frac{kg}{h} \cdot (1940\frac{J}{Kg \cdot K} \cdot (140°C - 100°C) + 2257 \cdot 10^3 \frac{J}{Kg} + \\
&+ 4186\frac{J}{Kg \cdot K} \cdot (100°C - 72.7°C)) = \\
&= 1.68 \cdot 10^8 \frac{J}{h} = 46.67kW
\end{aligned}
\tag{35}
$$

As such, the heat that will have to be supplied to the second column via both direct steam and the reboiler will be:

$$Q_{Col2,Tot} = Q_{Reb2} - Q_{DirectSteam} = 188.89kW - 46.67kW = 142.22kW \tag{36}$$

With this, the total amount of energy that has to be provided by the two reboilers via heat exchangers becomes

$$Q_{R,Tot} = Q_{Reb1} + Q_{Col2,Tot} = 569.44kW + 142.22kW = 711.66kW \tag{37}$$

In order to better evaluate the energy expenditure, it's possible to calculate the energy used per kilogram of feed and per kilogram of desired product.The results are available in table 15

Table 14: Energy required for the production of grappa

	Total energy [kW] [kw]	Energy per kg of feed [kWh/kg$_{\text{Feed}}$]	Energy per kg of product [kWh/kg$_{\text{Prod}}$]
Cooling	654.70	0.45	1.95
Heating	711.66	0.49	2.12

Another way to evaluate the energy costs is to express the energy needed in terms of utilities (cold water and steam), assuming a 100% efficiency on the heat exchangers. To calculate the amoung of cold water needed for cooling, equation 38 can be used:

$$\begin{aligned}
\dot{W}_{W,Uti} &= \sum_i \frac{|Q_{C,i}|}{c_{P,W} \cdot (T_{Out} - T_{In})} = \\
&= \frac{4.13 \cdot 10^7 \frac{J}{h}}{4186 \frac{J}{kg \cdot K} \cdot (19 - 15.3)°C} + \frac{482.2 \cdot 10^9 \frac{J}{h}}{4186 \frac{J}{kg \cdot K} \cdot (65 - 15.3)°C} + \\
&+ \frac{161 \cdot 10^8 \frac{J}{h}}{4186 \frac{J}{kg \cdot K} \cdot (52.9 - 15.3)°C} = \\
&= 14550 \frac{kg}{h} = 14.55 \frac{m^3}{h}
\end{aligned} \tag{38}$$

For these reboilers, the mass of steam needed to provide the required heating can be calculated with:

$$\begin{aligned}
\dot{W}_{Steam,Uti} &= \frac{Q_{R,Tot}}{c_{P,Steam} \cdot (T_{In} - T_{Boil}) + C_{V,W} \cdot 10^3} = \\
&= \frac{711.66kW \cdot 3.6 \cdot 10^6 \frac{J}{kW \cdot h}}{1940 \frac{J}{kg \cdot K} \cdot (140°C - 100°C) + 2257 \cdot 10^3 \frac{J}{kg}} = \\
&= 1097.4 \frac{kg}{h}
\end{aligned} \tag{39}$$

It should also be taken into consideration that the first column's residue stream, before being sent to the waste water treatment plant, enters an heat exchanger shell-side in order to heat up the phlegm entering tube-side from room temperature to around 45°C, thus reducing the energy duty of the reboiler by about 31.4 kW.

Chapter 5 - Possible improvements to the plant

5.1 Energy balance with partial condenser

Since stream Dist1 is also the feed stream of the second column, using a total condenser for the first column leads to a waste of energy condensing the vapor and then heating it back up past the boiling point within the second column's reboiler. As such, a partial condenser would allow to keep the distillate stream in saturated vapor phase and sparing the energy consumption at the reboiler. Equations 40, 41 and 42 show the theoretical energy savings for cooling and heating respectively.

$$\begin{aligned}
Q_{C1,Savedl} = &(c_{P,W} \cdot \dot{V}_{D1} \cdot \rho_{D1} \cdot x_{W,D1} + \\
&+ c_{P,Et} \cdot \dot{V}_{D1} \cdot \rho_{D1} \cdot x_{Et,D1} + \\
&+ c_{P,Met} \cdot \dot{V}_{D1} \cdot \rho_{D1} \cdot x_{Met,D1} + \\
&+ c_{P,EA} \cdot + \dot{V}_{D1} \cdot \rho_{D1} \cdot x_{EA,D1}) \cdot (T_{C,Out} - T_{C,In}) + \\
&+ (-c_{V,W} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{W,D1} - \\
&- c_{V,Et} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{Et,D1} + \\
&- c_{V,Met} \cdot (\dot{V}_{Ref1} + \dot{V}_{D1}) \cdot \rho_{D1} \cdot x_{Met,D1}) = \\
&= (4186 \frac{J}{kg \cdot K} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.091 + \\
&+ 2438 \frac{J}{kg \cdot K} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.885 + \\
&+ 2512 \frac{J}{kg \cdot K} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.016 + \\
&+ 1904 \frac{J}{kg \cdot K} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.006) \cdot (45 - 76)^\circ C + \\
&+ (-2257 \frac{kJ}{kg} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.091 + \\
&- 855 \frac{kJ}{kg} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.885 + \\
&- 1098.3 \frac{kJ}{kg} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.016) + \\
&- 404 \frac{kJ}{kg} \cdot 340 \frac{dm^3}{h} \cdot 0.8164 \frac{kg}{dm^3} \cdot 0.006) = \\
&= -2.95 \cdot 10^8 \frac{J}{h} = -81.94 kW
\end{aligned} \tag{40}$$

Assuming $\varphi = 0$ (saturated vapor conditions) for stream Dist1, the calculation of the energy at the reboiler is:

$$\begin{aligned}
Q_{Reb2,SatVap} &= -Q_{21} - \lambda_2 \cdot \varphi_2 \cdot \dot{X}_{D1} = \\
&= 5.80 \cdot 10^8 \frac{J}{h} = 161.11 kW
\end{aligned} \tag{41}$$

That is equal to an energy saving of:

$$Q_{Reb2,Saved} = Q_{Reb2} - Q_{Reb2,SatVap} = 3.06 \cdot 10^8 \frac{J}{h} = 27.78 kW \tag{42}$$

These values can be scaled in regards to the feed and the product flow rates, just like in table 15

Table 15: Energy saved with partial condenser

	Total energy [kW] [kw]	Energy per kg of feed [kWh/kg_{Feed}]	Energy per kg of product [kWh/kg_{Prod}]
Cooling	81.94	0.06	0.24
Heating	27.78	0.02	0.08

Converting the values obtained from equations 40 and 42 into utilities' demand, as done with equations 38 and 39, and assuming the same outlet temperatures are to be kept for the utlities the results are calculated in equation 43

$$\begin{aligned} \dot{W}_{W,UtiSaved} &= \frac{|Q_{C,Saved}|}{c_{P,W} \cdot (T_{Out} - T_{In})} = \frac{2.95 \cdot 10^8 \frac{J}{h}}{4186 \frac{J}{kg \cdot K} \cdot (65 - 15.3)°C} = \\ &= 1355.25 \frac{kg}{h} = 1.36 \frac{m^3}{h} \\ \dot{W}Steam, UtiSaved &= \frac{Q_{Reb2,Saved}}{c_{P,Steam} \cdot (T_{In} - T_{Out}) + C_{V,W} \cdot 10^3} = \\ &= \frac{3.02 \cdot 10^8 \frac{J}{h}}{1940 \frac{J}{kg \cdot K} \cdot (140°C - 100°C) + 2257 \cdot 10^3 \frac{J}{kg}} = \\ &= 129.36 \frac{kg}{h} \end{aligned} \tag{43}$$

5.2 Sizing of an additional column for ethanol recovery from wastes

Removing methanol from the top of the second column requires some amount of ethanol to be withdrawn from there as well, thus wasting some product (2.62 $\frac{kg}{h}$ according to table 7). As such, designing a third column to further separate methanol and ethanol in the Dist2 stream could increase the overall plant efficiency.

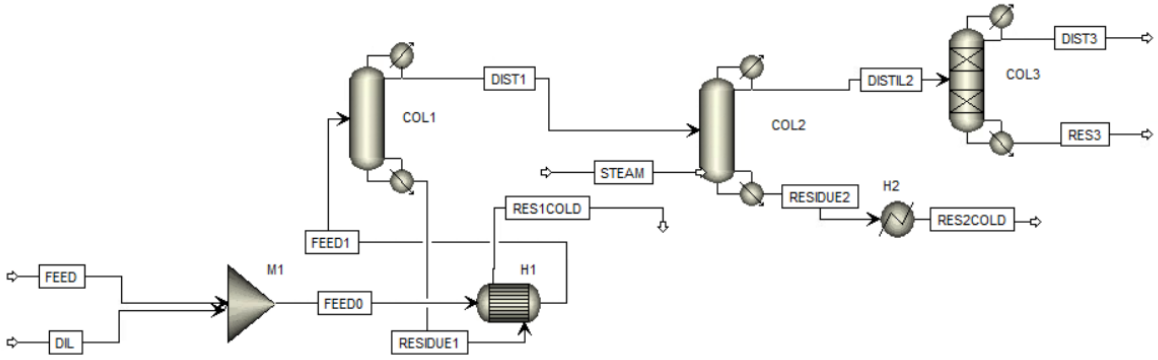

Figure 10: Representation of the distillation train with the third column for ethanol recovery

The column will be designed under some assumptions: Firstly, 2-butanol, propanol and isobutanol will be considered non-existant within the stream, instead of using the negative values calculated via mass balances. Secondly, the mass fraction of methanol within the residue stream of this new column will be chosen to be the same as the mass fraction of methanol in the Res2 stream, in order to keep the quality of the product consistent, while the molar fraction of methanol in the distillate will be set to 0.8 to recover most of the ethanol while not adding too many trays to the column. Lastly, the amount of 1-butanol is relatively small compared to the other compounds, so it will be ignored for the sake of simplicity. On the other hand, the molar fraction of acetaldehyde and ethyl-acetate will be considered to be zero within the new column's residue stream, while water's molar fraction will be considered zero in the distillate stream.

The first step to design a distillation column is to calculate the mass fractions of the components to be separated and the mass flow rates of the distillate and residue streams. That can be accomplished by solving the system

$$\begin{cases} \dot{W}_{Et,Dist2} + \dot{W}_{Met,Dist2} = \dot{W}_{Dist3} + \dot{W}_{Res3} \\ (\dot{W}_{Et,Dist2} + \dot{W}_{Met,Dist2}) \cdot x_{Met,Dist2} = \dot{W}_{Dist3} \cdot x_{Met,Dist3} + \dot{W}_{Res3} \cdot x_{Met,Res3} \end{cases} \tag{44}$$

$$\begin{cases} 2.62\frac{kg}{h} + 3.53\frac{kg}{h} = \dot{W}_{Dist3} + \dot{W}_{Res3} \\ (2.62\frac{kg}{h} + 3.53\frac{kg}{h}) \cdot \frac{0.110}{0.110+0.057} = \dot{W}_{Dist3} \cdot 0.8 + \dot{W}_{Res3} \cdot 0.004 \end{cases}$$

Whose results are:

$$\begin{cases} \dot{W}_{Dist3} = 4.40\frac{kg}{h} \\ \dot{W}_{Res3} = 1.75\frac{kg}{h} \end{cases} \tag{45}$$

To these results, the mass flow rates of acetaldehyde, ethyl-acetate and water to the respective stream, as detailed earlier. The final values are $\dot{W}_{Dist3} = 6.26\frac{kg}{h}$ and $\dot{W}_{Res3} = 3.35\frac{kg}{h}$. The calculated mass flow rates of streams Dist2, Dist3 and Res3 are reported in table 16 below and the results transposed in molar flow rates and mass fractions in tables 17 and 18.

Table 16: Mass flow rates for column 3 in [kg/h]

	Dist2	Dist3	Res3
Acetaldehyde	0.17	0.17	0.00
Ethyl-acetate	1.69	1.69	0.00
Methanol	3.53	3.52	0.01
Ethanol	2.62	0.88	1.64
Water	1.60	0.00	1.60

Table 17: Molar flow rates for column 3 in [kmol/h]

	Dist2	Dist3	Res3
Acetaldehyde	0.00	0.00	0.00
Ethyl-acetate	0.02	0.02	0.00
Methanol	0.11	0.11	0.00
Ethanol	0.06	0.02	0.04
Water	0.09	0.00	0.09

Table 18: Mass fractions for column 3

	Dist2	Dist3	Res3
Acetaldehyde	0.02	0.03	0.00
Ethyl-acetate	0.18	0.27	0.00
Methanol	0.37	0.56	0.00
Ethanol	0.27	0.14	0.50
Water	0.17	0.00	0.49

With these data, it's possible to calculate the boiling temperature of the mixture, its molar weight and its density in both liquid and vapor phases, as shown below:

$$\begin{aligned} log_{10}(760) &= \sum_i [\gamma_i \cdot X_i \cdot (A_i - \frac{B_i}{T_{Boil,D2} + C_i})] \\ T_{Boil,D2} &= 76.36°C \\ MW_{D2} &= \sum_k (MW_k \cdot x_k) = 34.45 \frac{g}{mol} \\ \rho_{L,Col3} &= \sum_k (\rho_k \cdot x_k) = 0.844 \frac{kg}{dm^3} \\ \rho_{V,Col3} &= \frac{MW_{D2} \cdot P}{R_{Gas} \cdot T_{Boil,D2}} \cdot \frac{1}{1000} = 0.001 \frac{kg}{dm^3} \end{aligned} \tag{46}$$

The flow rate of the reflux has been chosen to be double the flow rate of the feed, as is industrial standard for demethylation columns. In addition, the feed will be considered as saturated vapor, meaning that the condenser for column 2 will be considered to be a partial condenser, and that will be taken into consideration when calculating the energy needed for the third column.

With these data, McCabe-Thiele construction can be used in order to evaluate the number of ideal stages for the separation:

$$
\begin{aligned}
\dot{W}_{L,Col3} &= 2 \cdot \dot{W}_{Dist2} = 15.51 \frac{kg}{h} \\
\dot{W}_{V,Col3} &= \dot{W}_{L,Col3} + W_{Dist3} = 21.77 \frac{kg}{h} \\
r &= \frac{\dot{W}_{L,Col3}}{W_{Dist3}} = 2.477 \\
\varphi_{Dist2} &= 0 \\
F_{L,V} &= \frac{\dot{W}_{L,Col3}}{\dot{W}_{V,Col3}} \cdot \sqrt{\frac{\rho_V}{\rho_L}} = 0.0269
\end{aligned}
\tag{47}
$$

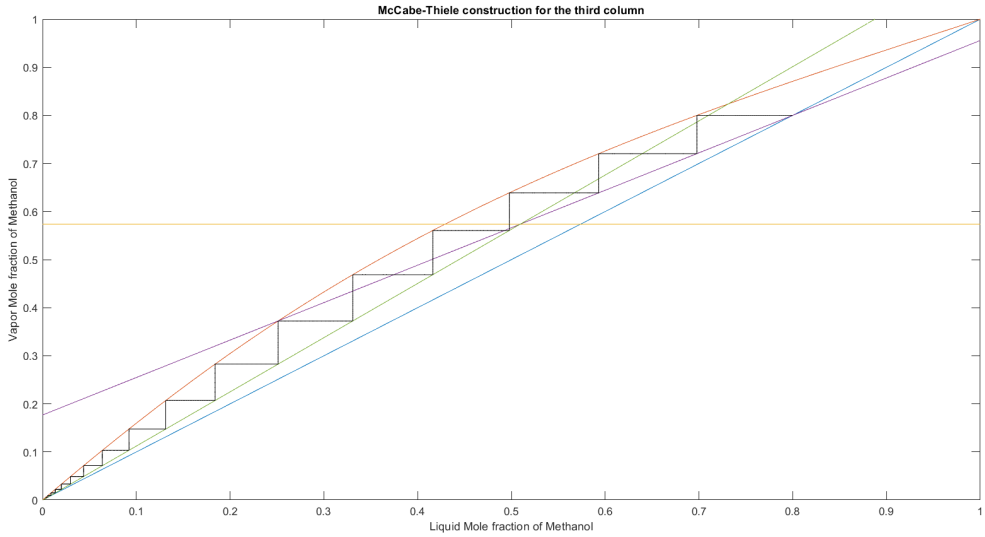

Figure 11: McCabe-Thiele construction for Column 3

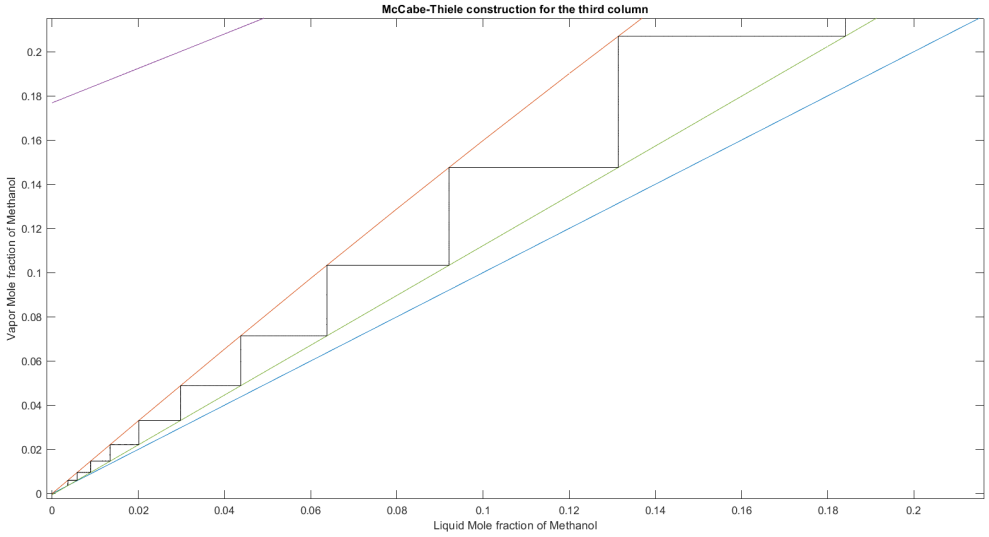

Figure 12: Zoom of McCabe-Thiele construction for Column 3 at the end of the stripping section

5.2.1 Tray column sizing

In order to size a tray column, a first guess of the diameter has to be used. Here, a value of 0.15 m has been chosen. From this, the distance between trays can be estimated using the empirical correlation

$$H_{Guess} = 0.41 \cdot \sqrt{D_{Guess}} = 0.16m \tag{48}$$

Using Fair's diagram, it's possible to estimate the capacity factor at flooding, as shown in figure 13:

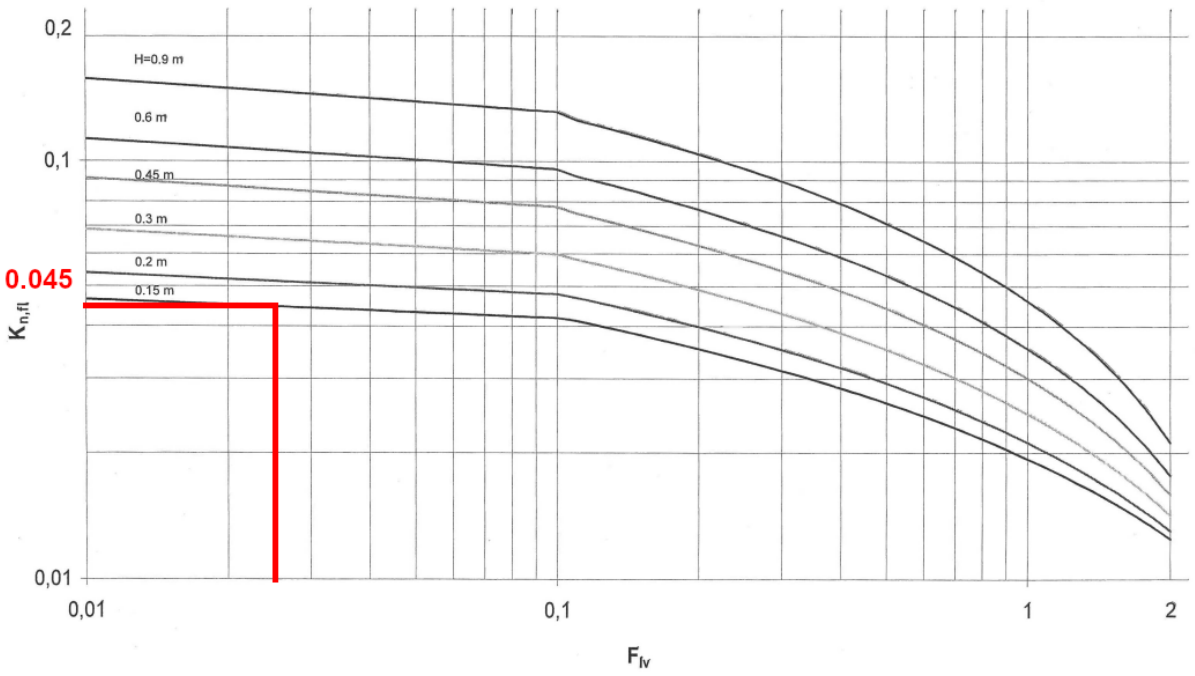

Figure 13: Estimation of capacity factor at flooding for tray column

The formula to calculate the flooding velocity is

$$u_{n,fl} = K_{n,fl} \cdot K_1 \cdot K_2 \cdot K_3 \cdot K_4 \cdot \sqrt{\frac{\rho_L - \rho_V}{\rho_V}} \tag{49}$$

where

$$\begin{aligned} K_1 &= (\frac{\sigma}{20})^{0.2} = (\frac{20}{20})^{0.2} = 1 \\ K_2 &= (10 \cdot \frac{A_{Holes}}{A_{Active}})^{0.44} = 1 \text{ assuming } \frac{A_{Holes}}{A_{Active}} = 0.1 \\ K_3 &= 1 \text{ assuming holes smaller than 6mm in diameter} \\ K_4 &= 1 \text{ no foaming in the system} \end{aligned} \tag{50}$$

Equation 49 reduces to $u_{n,fl} = K_{n,fl} \cdot \sqrt{\frac{\rho_L - \rho_V}{\rho_V}} = 1.19 \frac{m}{s}$

Assuming a safety factor $K_5 = 0.8$, the vapor velocity will be $u_n = K_5 \cdot u_{n,fl} = 0.8 * 1.19 = 0.95 \frac{m}{s}$.

The next step is to calculate the net area of the tray using equation 51:

$$A_n = \frac{\dot{V}_V}{u_n} = \frac{\dot{W}_V}{\rho_V \cdot u_n} = \frac{21.77 \frac{kg}{h}}{0.001 \frac{kg}{dm^3} \cdot 1000 \frac{dm^3}{m^3} \cdot 3600 \frac{s}{h} \cdot 0.95 \frac{m}{s}} = 6.4 \cdot 10^{-3} m^2 \tag{51}$$

That is equivalent to a diameter of

$$D = \sqrt{\frac{4 \cdot A}{\pi}} = 0.09m = 9cm \tag{52}$$

While this calculation doesn't take into account the difference between net area and total area, thus being a bit lower than the expected diameter when taking into account the downcomers, its value is too low to take into consideration the possibility of building a tray colum, as a diameter of around 25 cm is usually recommended[11]. As such, a tray column is not recommended, and packed columns should used instead.

5.2.2 Random packing column sizing

Using Eckert's diagram, it's possible to find the capacity factor at flooding of a random packed column using the flux parameter F_{lv} calculated previously

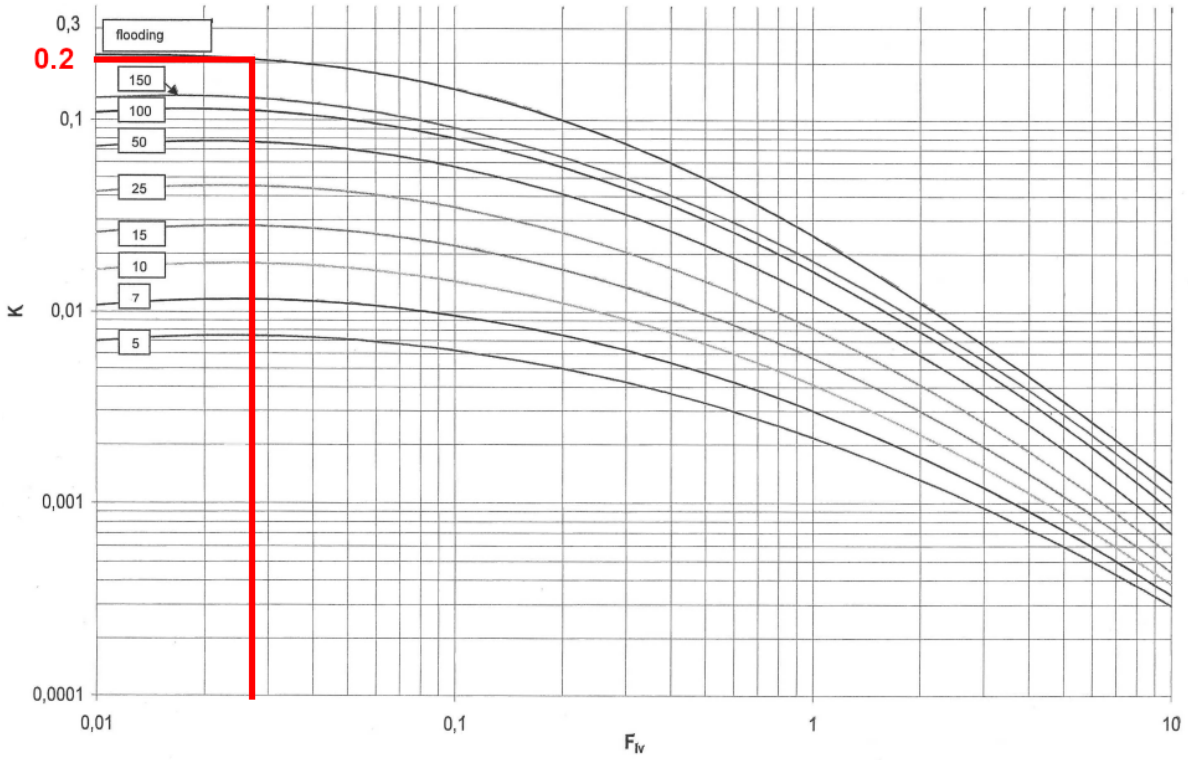

Figure 14: Estimation of capacity factor at flooding for packed column

The correlation to calculate the vapor flooding velocity once the capacity factor at flooding is known is shown in equation 53

$$(\frac{V_p}{A})_{fl} = \sqrt{\frac{K_{fl} \cdot g \cdot \rho_V \cdot \rho_L}{C_f \cdot (\frac{\mu_L}{\mu_W})^{0.2} \cdot (\frac{\rho_W}{\rho_L})}} \tag{53}$$

In order to calculate the mass flooding velocity, a random packing has to be chosen and data about the mixture have to be calculated.

For the packing, 13 mm ceramic Raschig rings have been chosen. According to literature [11] $C_f = 2000\frac{1}{m}$.

The viscosity of the mixture can be calculated as

$$\mu_L = \sum_k w_k \cdot \mu_k = 7.91 \cdot 10^{-4} Pa \cdot s \tag{54}$$

The flooding mass velocity can thus be calculated

$$(\frac{V_p}{A})_{fl} = \sqrt{\frac{0.2 \cdot 9.81\frac{m}{s^2} \cdot 1\frac{kg}{m^3} \cdot 844\frac{kg}{m^3}}{2000\frac{1}{m} \cdot (\frac{7.91 \cdot 10^{-4} Pa \cdot s}{1 \cdot 10^{-3} Pa \cdot s})^{0.2} \cdot (\frac{0.001\frac{kg}{dm^3}}{0.844\frac{kg}{dm^3}})}} = 0.938\frac{Kg}{m^2 \cdot s} \tag{55}$$

Choosing a safety factor $K_5 = 0.8$, it's possible to calculate the column's section as

$$A = \frac{\dot{W}_V}{K_5 \cdot (\frac{V_p}{A})_{fl}} = \frac{21.77\frac{kg}{h}}{0.8 \cdot 0.938\frac{Kg}{m^2 \cdot s} \cdot 3600\frac{s}{h}} = 0.0081 m^2 \tag{56}$$

Which translates to a diameter of 10 cm. According to literature[12], a ratio of at least 10 between the column's diameter and the packing diameter is necessary to have a good vapor-liquid interface. In this case, the ratio is 7.69, which is too low, and leads to discarding the idea of a random packing column.

5.2.3 Structured packing column sizing

Similarly to the random packing column, the choice of packing is important for the structured packing too. Referencing literature [13], plastic Mellapack 250Y from Sulzer have been chosen $C_f = 72\frac{1}{m}$.

In order to estimate the capacity parameter, the Generalized Pressure Drop Correlation proposed by Kister and Gill has been used.

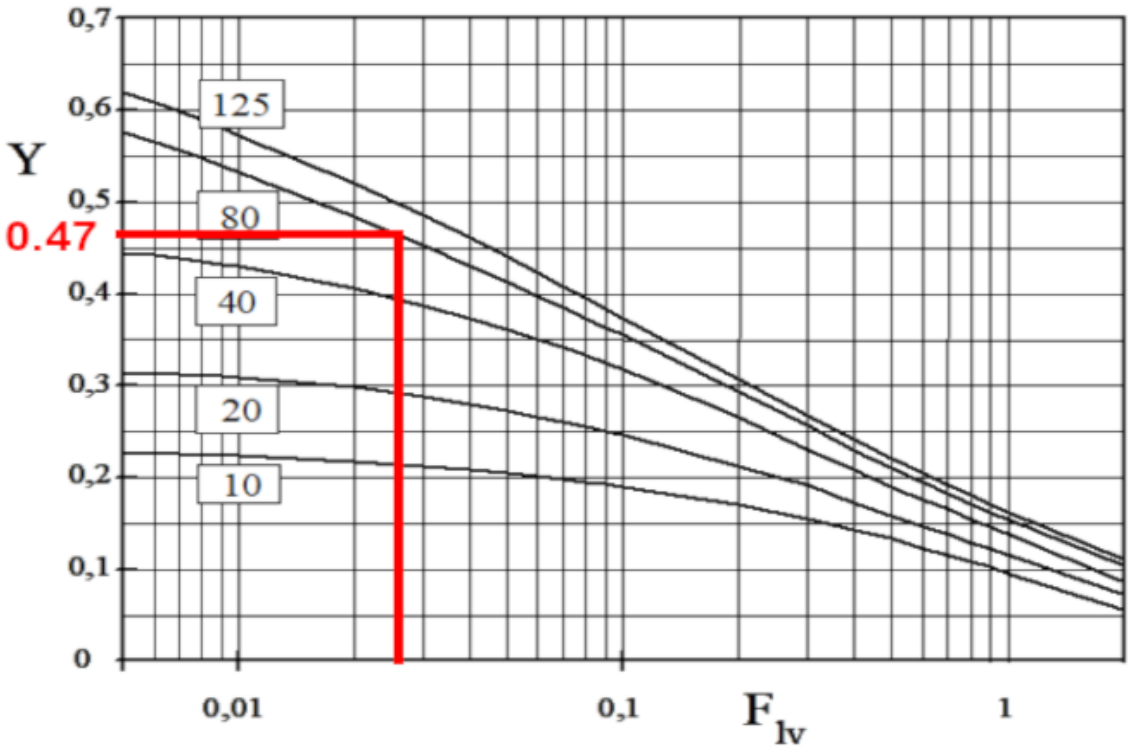

Figure 15: Generalized Pressure Drop Correlation proposed by Kister and Gill

The flooding curve has been chosen by using equation 57

$$(\frac{\Delta P}{L})_{fl} = 4.17 \cdot C_f^{0.7} = 83\frac{mmH_2O}{m} \tag{57}$$

From that, the value of Y corresponding the value of $Y_{fl} = 0.47$ has been found. The value of vapor flooding velocity can be calculated with equation 58

$$u_{f,lv} = \frac{Y_{fl}}{C_f^{0.5} \cdot \nu^{0.05}} \cdot \sqrt{\frac{\rho_L - \rho_V}{\rho_V}} = 2.92\frac{kg}{m^2 \cdot s} \tag{58}$$

The column's section can be calculated, assuming a safety factor $K_5 = 0.8$ as

$$A = \frac{\dot{W}_V}{K_5 \cdot u_{f,lv}} = \frac{21.77\frac{kg}{h}}{0.8 \cdot 2.92\frac{Kg}{m^2 \cdot s} \cdot 3600\frac{s}{h}} = 0.0026m^2 \tag{59}$$

Which translates to a diameter of 5.7 cm. Rounding it up to 6cm, it's necessary to calculate the vapor velocity to make sure it's under 80% of the flooding velocity

$$u_v = \frac{V_p}{A} = \frac{21.77\frac{kg}{h}}{3600\frac{s}{h} \cdot (\frac{0.06^2 \cdot \pi}{4})m^2} = 2.14\frac{kg}{m^2 \cdot s} \tag{60}$$

$$\frac{u_v}{u_{f,lv}} = \frac{2.14}{2.92} = 0.73 < 0.80 \tag{61}$$

As visible on figure 11, the number of stages necessary for this separation is 20. In literature [13], an HETP of 0.38 m per stage is reported. As such, the height of the column can be calculated by multiplying the HETP by the number of stages, reaching an height of 7.60 m. The pressure drop can be estimated with equation 57, multiplying the value of Y found by the height of the column, resulting in a pressure drop of $632.5 mm H_2O$.

5.2.4 Energy balance

The energy saved from using a partial condenser for the second column can be estimated with

$$\begin{aligned} Q_{C3} &= (c_{P,W} \cdot \dot{W}_W + c_{P,Et} \cdot \dot{W}_{Et} + c_{P,Met} \cdot \dot{W}_{Met} + c_{P,EA} \cdot \dot{W}_{EA}) \cdot (T_{C,Out} - T_{C,In}) \\ &\quad - (C_{V,W} \cdot \dot{W}_W + C_{V,Et} \cdot \dot{W}_{Et} + C_{V,Met} \cdot \dot{W}_{Met} + C_{V,EA} \cdot \dot{W}_{EA}) = \\ &= 4.33 \cdot 10^5 \frac{J}{h} = 0.12kW \end{aligned} \tag{62}$$

Assuming a similar exit temperature to that of Cond2 in the current plant, the energy at the condenser can be calculated with

$$\begin{aligned} Q_{C3} &= (c_{P,Et} \cdot \dot{W}_{Et} + c_{P,Met} \cdot \dot{W}_{Met} + c_{P,EA} \cdot \dot{W}_{EA}) \cdot (T_{C,Out} - T_{C,In}) \\ &\quad - (C_{V,Et} \cdot \dot{W}_{Et} + C_{V,Met} \cdot \dot{W}_{Met} + C_{V,EA} \cdot \dot{W}_{EA}) = \\ &= (2438\frac{J}{kg \cdot K} \cdot 0.88\frac{kg}{h} + 2512\frac{J}{kg \cdot K} \cdot 3.52\frac{kg}{h} + 1904\frac{J}{kg \cdot K} \cdot 1.69\frac{kg}{h}) \cdot (45 - 62.2)^\circ C) + \\ &\quad + (-855\frac{kJ}{kg} \cdot 0.88\frac{kg}{h} - 1098.3\frac{kJ}{kg}3.52\frac{kg}{h} - 404\frac{kJ}{kg} \cdot 1.69\frac{kg}{h}) = \\ &= -51.55 \cdot 10^6 \frac{J}{h} = -1.54kW \end{aligned} \tag{63}$$

Since $\varphi = 0$ for the vapor entering the third column, the energy needed will be equal to that at the condenser, but with opposite sign, so $Q_{R3} = 1.54kW$.

These can be converted to usage of cold water and steam respectively, to $0.03\frac{m^3}{h}$ and $2.37\frac{kg}{h}$.

Conclusions

In this thesis, the performance of a distillation plant, located in Visná di Vazzola, for the production of grappa was assessed and discussed.

Data about the flow rates of the various streams and their compositions were gathered from samples taken from the plant at steady state. Stream compositions and flow rates for the other streams were calculated via mass balances after converting the compositions into mass flow rates, taking into account the volume contraction due to water and ethanol mixing. The calculated values were used to perform McCabe-Thiele construction in order to calculate the ideal number of stages and compare it with the real number of stages to find the two columns' efficiency.

The energy duty of the distillation train has been calculated assuming a 100% efficiency of the heat exchangers and, as a point of improvement, the energy saving resulting from a partial condenser has also been calculated.

The results obtained from the mass balances are satisfactory and within the error margins of the instrumentation used. The negative values obtained are small compared to the other values and can be attributed to the instrumentation or to a non-homogeneity in the system, as phlegm might not be perfectly mixed before entering the distillation column. The results in table 7 are close to what was expected, so the method used and the calculations done are appropriate.

The calculated efficiency of the two colums in the cases analyzed is low, but it's hard to improve without impacting the quality of the final product in the case of the first column, or without losing the possibility to adapt to a different amount of light components to separate in the case of the second column. In addition, a small error in the measurement of density of the Dist1 stream has an high impact on the calculated number of ideal stages due to the closeness to the azeotrope between water and ethanol. In short, it might be possible to increase the efficiency of the columns, but the trade-off would be a possible loss in quality.

Switching to a partial condenser for Column1 would lead to a substantial decrease in energy consumption and it might be worth the investment. However, mass flow rate would be more challenging to control and would require a redesign of the control system on the reflux flow rate.

Lastly, the implementation of a third column to recover part of the ethanol present the Dist2 stream was analyzed. The design of this column was developed, however the convenience of adding it would need to go through a cost analysis, taking into account both the capital cost and the operative costs, versus the potential revenue coming from more product. It should be noted how the energy costs related to the third column are almost negligible compared to the rest of the plant.

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