



Recommissioning of a dividing-wall column pilot plant according to Kaibel's design and corresponding experiments.

Wiederinbetriebnahme einer Pilotanlage einer Trennwandkolonne nach dem Design von Kaibel und entsprechende Experimente

Research project

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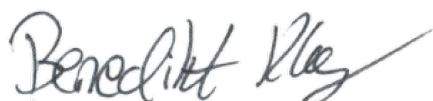
City, Date Trondheim, November 2, 2023

Affidavit

I hereby declare in lieu of oath that I prepared the present work independently. Ideas adopted from external sources directly or indirectly are labeled accordingly.

This thesis was not submitted to another examination board.

Trondheim, November 2, 2023

A handwritten signature in black ink, reading "Benedikt Kloss". The signature is written in a cursive style with a long horizontal stroke at the end. It is positioned above a solid horizontal line.

Benedikt Kloss, B.Sc.

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Symbols

Latin Symbols

A	Area	m^2
D	Distillate flow	$\frac{\text{m}^3}{\text{s}}$
F	Feed flow	$\frac{\text{L}}{\text{s}}$
I	integral time constant	-
K_p	controller gain	-
L	Liquid reflux flow	$\frac{\text{m}^3}{\text{s}}$
m	mass	kg
N	Number of components	-
p	Pressure	Pa
Q	reboiler duty	W
q	liquid fraction of feed	-
R_L	Liquid split ratio	-
R_V	Vapor split ratio	-
S_1	upper side stream	$\frac{\text{m}^3}{\text{s}}$
S_2	lower side stream	$\frac{\text{m}^3}{\text{s}}$
T	Temperature	K
u	Controller output	-
u_D	Output of controller D	-
u_{S1}	Output of controller S_1	-
u_{S2}	Output of controller S_2	-
V	Vapor flow	$\frac{\text{m}^3}{\text{s}}$
w	Mass fraction	-
x	Molar fraction in liquid phase	-
y	Molar fraction in gaseous phase	-
z	Molar fraction of components	-

Greek Symbols

α	relative Volatility	-
Δ	Difference	-

ρ	Density	$\frac{\text{kg}}{\text{m}^3}$
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Subscript Indices

i	Component i
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1 Introduction

The present work was carried out in the course of a research internship at the Department of Chemical Engineering at the Norwegian University of Science and Technology in Trondheim. This research internship as well as the present thesis were part of the author's Master's degree in Chemical Engineering at the Technical University of Munich.

Distillation is one of the most important separation operations in today's industry. It is used to separate binary as well as multi-component liquid mixtures. This process is mostly used in petrochemical and chemical industries. Distillation is performed in so-called distillation columns.

Distillation has a high energy demand. Therefore it is desirable to minimize the energy consumption of this process for economical and environmental reasons. A possibility to reduce the energy consumption of the separation of multi-component mixtures via distillation is the usage of so-called dividing wall columns.

The separation of multi-component mixtures conventionally requires a sequence of several binary distillation columns. Dividing wall columns represent an one-shell arrangement for a sequence of thermally coupled columns to separate multi-component mixtures. With the use of dividing wall columns, up to 30 % of energy can be saved compared to a classical sequence of columns. [LI ET AL. 2020] Additionally, investment costs can be reduced by using dividing wall columns.

A special form of a dividing wall column is the so-called Kaibel column. This arrangement was proposed by KAIBEL 1987. With this column variant a four-component mixture can be separated using only a single dividing wall. Accordingly, the Kaibel design offers the possibility of further simplification in terms of equipment as well as energy saving potential.

The challenge lies in optimal operation of dividing wall columns since the energy saving potential is lost by operating out of the optimum. Therefore an adequate control structure of the column is necessary to operate the column at the optimum operating point. This allows the full energy-saving potential to be exploited.

During the doctoral thesis of STRANDBERG 2011 a pilot-scale Kaibel column was constructed and put into operation at the Department of Chemical Engineering at the Norwegian University of Science and Technology in Trondheim to study optimal operation and control of dividing wall columns. This column is the only one in the world that has an active vapor split as a manipulable variable. DWIVEDI 2013 investigated control structures for the Kaibel column using a vapor split control in his doctoral thesis.

The mentioned Kaibel pilot plant had not been in operation for some time. For this reason, the goal of the research internship, in the course of which this thesis was written,

was to recommission and improve the Kaibel column. At the beginning, the control software in *LabView* was updated and small improvements were added. After several small maintenance works at the column, the pilot plant was put back into operation. Only a three-component mixture was used, as the harmful methanol previously used as the fourth component was no longer to be used. At the end several experiments using various control schemes were conducted.

Due to the time limitation of the internship, not every possible improvements and experiments could be carried out. Thus, the vapor split valves could not be optimized in the time available, although there is potential for improvement in this regard. In addition, not all experiments could be validated by reproduction measurements.

A brief description of the theory of distillation and, in particular, of dividing wall columns is given in the first chapter. In addition, the control of dividing wall and Kaibel columns is described. In the second chapter, the experimental setup of the Kaibel pilot plant is described and a description of the experimental procedure is given. In the following chapter, the conducted experiments are described and the results are discussed. A short summary and an outlook is given at the end of the thesis.

2 Theoretical background and state of the art

In this the chapter the general basics of distillation are described briefly. Further the fundamentals of dividing wall columns as well as the state of the art are presented.

2.1 Fundamentals of distillation

Distillation is a common method to separate liquid mixtures into their components. It is used especially in petrochemical and chemical related industries. Distillation is known for a very long time, since the ancient Greeks already used it. [STICHLMAIR ET AL. 2021] The principle of distillation is the same as of every other thermal separation method. The principle is characterized by three steps. The first step is the generation of two different phases. These two phases have different compositions. In a second step a intimate contact between the two phases is created. This means that the phase interface should be maximized as far as possible. Since the two phases are not in a thermodynamic equilibrium, mass transfer between the phases occur. In the third and last step the two phases are separated again. [STICHLMAIR ET AL. 2021]

The figure 2.1 illustrates the described principle of separation.

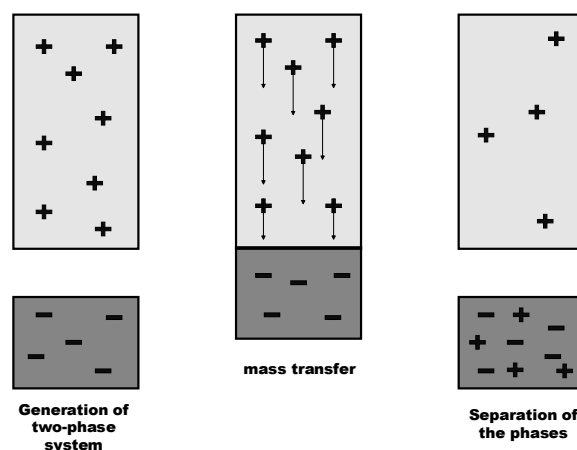


Figure 2.1: Three steps of separation principle

In addition to distillation, this principle is also used in absorption, extraction or adsorption. Thereby the two phases could be gas-liquid, liquid-liquid or liquid-solid. In the considered case of distillation the two-phase system consists of a gaseous and a liquid phase.

The gaseous phase during distillation is generated by partial vaporization of the liquid feed. Therefore distillation only requires heat instead of an auxiliary phase. Heat can be easily removed. This is an advantage of distillation against other separation methods. [STICHLMAIR ET AL. 2021]

These two phases are brought into intensive contact. Therefore mass transfer between the phases occurs. In the case of a binary mixture, the light-boiling component accumulates in the gas phase while the heavy boiling component passes into the liquid phase. The driving force of the mass transfer is the difference between the actual concentrations and the equilibrium concentrations of the substances. It has to be ensured that this difference is sufficiently large during operation. [STICHLMAIR ET AL. 2021]

Subsequently the two phases are separated again. The resulting liquid consists of a higher amount of high boiling component and the gas consists mainly of light component. This is called a single-stage distillation. The gaseous phase is eventually condensed afterwards. A single-stage distillation achieves only limited purity.

2.1.1 Practical implementation of multi-stage distillation

In order to achieve high purities, which are mostly required in the industry, a multi-stage distillation is applied. The multi-stage distillation can be considered as a combination of several single stages. The principle of multi-stage distillation is illustrated in figure 2.2. The dotted lines represent gaseous streams and the solid lines represent liquid streams.

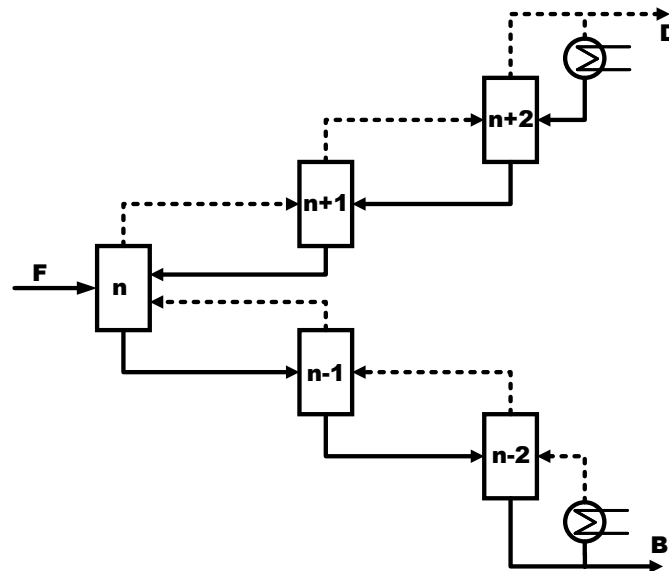


Figure 2.2: Schematic illustration of multi-stage distillation

The top product of the first distillation is subjected as feed to a second distillation. The top product of the second stage is subjected to the third distillation and so on. The resulting bottom products of each stage are subjected as reflux to the previous stage. The bottom product of the first distillation is subjected in an equivalent way to several distillation stages. Thereby the respective overhead products are returned

as reflux. The two phases are brought into intense contact at each stage to improve mass transfer. Accordingly, the respective product streams are purified further and further through every stage. Through this sequence of distillation stages, the light boiler accumulates at the top end and the heavy boiler at the bottom end. A condenser at the top and a reboiler at the bottom of the sequence ensure the necessary reflux. [STICHLMAIR ET AL. 2021]

The single stages that are illustrated in figure 2.2 can be merged to one apparatus. The resulting apparatus is called distillation column. A column consists of several equilibrium stages. The fluids flow in countercurrent within the column and are brought into intensive contact on every stage. A schematic column is shown in figure 2.3.

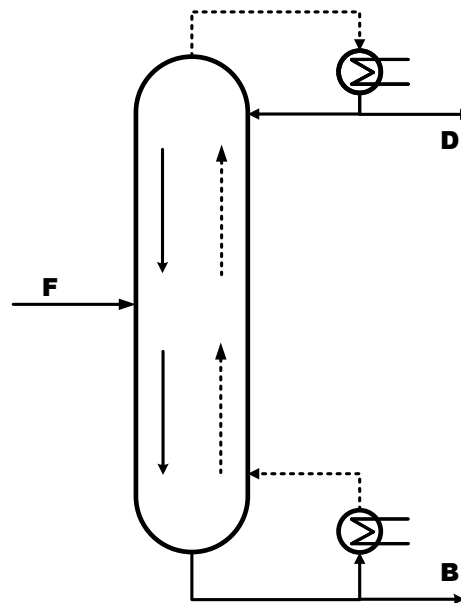


Figure 2.3: Schematic illustration of a distillation column

At the upper end of the column the product stream is condensed by a heat exchanger. One fraction of the top stream is returned as reflux to the column. The other part is drawn as product stream. Analogous, a fraction of the bottom stream is evaporated by a heat exchanger and returned to the column while the other fraction is drawn as bottom product stream. The part of the column above the feed inlet is called rectifying section. In this part the low boiling component accumulates. The part below the feed inlet is called stripping section. This is where the high boiling component accumulates. Inside the column different internals ensure the intensive contact between the phases and therefore the sufficient separation of the mixture. Depending on the mixture to be separated, different construction methods of column internals are used.

Trays and packings are the most common column internals. In a tray column, several trays are mounted on top of each other. The respective trays correspond to the stages of distillation. The gaseous phase is introduced into the liquid at the bottom of a tray through perforations and mixes with the liquid intensively. Thus, a two-phase layer with a large interfacial area is created in which the mass transfer occurs. The gas flows upwards in countercurrent to the liquid. The liquid is fed to a tray through a

downcomer and exits the tray over a weir into the next downcomer. To prevent liquid from flowing through the perforations to the lower tray, a certain gas flow must be ensured. [STICHLMAIR ET AL. 2021]

Depending on the application, different types of trays are used. The figure 2.4 shows examples of a Sieve tray, a Bubble cap tray and a Valve tray. These are the most important types of trays.

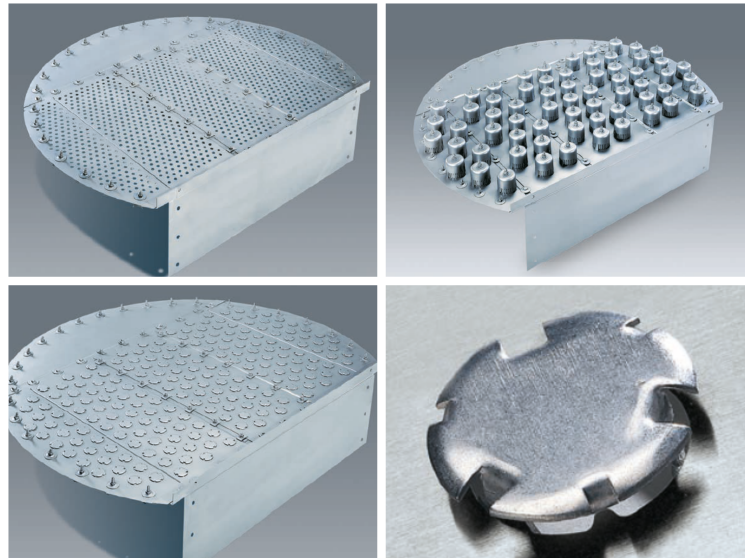


Figure 2.4: Examples of Sieve tray (upper left), Bubble cap tray (upper right) and Valve tray (bottom) [RVT 2022]

Inside of packed columns random or structured packings provide a large internal surface. On this surface the liquid can be distributed as a film. Thus a sufficient interfacial area between gas and liquid is ensured. Analogous to the tray column both phases flow in countercurrent inside a packed column. In this case the selection of the suitable packing type depends on the separation task as well. The figure 2.5 shows examples for random and structured packings.

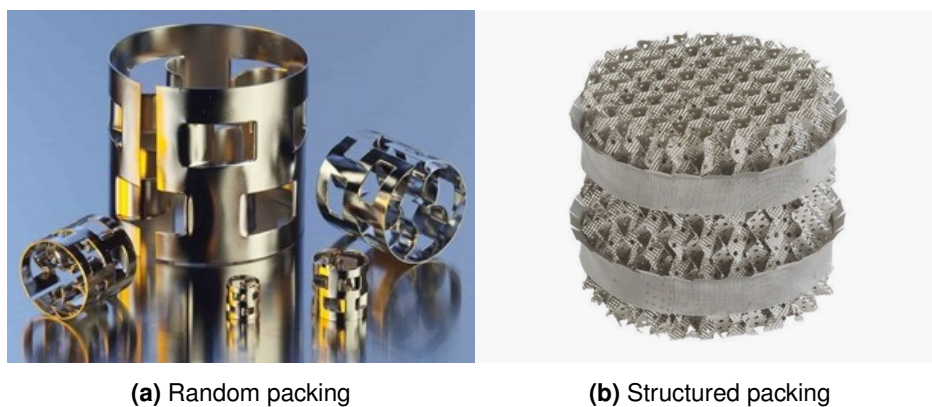


Figure 2.5: Examples of random and structured packings [SULZER 2023]

There are diverse shapes for particles used in random packings. Thereby saddles, rings and spheres are the most important basic shapes. These particles are stacked randomly

inside the column. Structured packings consist of different layers of metal sheets which are perforated and corrugated.

Uniform liquid distribution within the packing is crucial for the performance of packed columns. Therefore properly designed liquid distributors are necessary. With increasing run length of the liquid, the uneven distribution of liquid inside the packing increases as well. This is called maldistribution. Therefore the height of packed beds are limited and several liquid distributors may be necessary inside the column. [STICHLMAIR ET AL. 2021]

Packed columns prove to be advantageous over tray columns in terms of their low pressure drop. Whereas tray columns are less susceptible to fouling. Tray columns require a minimum gas flow, but can be operated at a variable liquid load. Packed columns, on the other hand, require a minimum liquid load to ensure packing wetting. However, in the case of packed columns, the gas load can be varied over a relatively wide range. [STICHLMAIR ET AL. 2021]

2.1.2 Separation of multiple component mixtures

So far only binary mixtures have been considered which are separable in a single column. Binary mixtures are an exceptional case in practical application. Most real applications in industry require the separation of multi-component mixtures.

The complete separation of a multiple component mixture demands several distillation steps. For the separation of a ternary mixture at least two sequential columns are necessary. [STICHLMAIR ET AL. 2021, ASPRION & KAIBEL 2010]

The figure 2.6 illustrates three different paths of separating a ternary mixture consisting of the components A, B and C into pure components. Where A has the lowest and C the highest boiling point.

The separation of a ternary mixture requires a sequence of columns. The most common configurations are the so called direct and indirect split. These two configurations are shown in figure 2.6a and 2.6b. During direct split the light boiling component A is separated in the first column. The remaining mixture of B and C is submitted to the second column and separated there. In the case of the indirect split the heavy boiling component C is separated first and the mixture of A and B is separated in the subsequent column. In this two configurations loss of efficiency due to formation of entropy of mixing and remixing of the intermediate boiling component is unavoidable. [WAIBEL ET AL. 2023]

The third configuration shown in figure 2.6c consists of three columns and is called the sloppy sequence. [DEJANOVIĆ ET AL. 2010] In this configuration the first column is called the prefractionator. In this first column a split between A and C is performed. The intermediate boiling component B distributes to both product streams. Both product streams are each fed to subsequent columns. The over head product stream of the prefractionator is separated into the pure components A and B. Hereby A is the over head product and B the bottom product of the subsequent column. Analogous the bottom product stream of the prefractionator is separated into pure B and C. In this

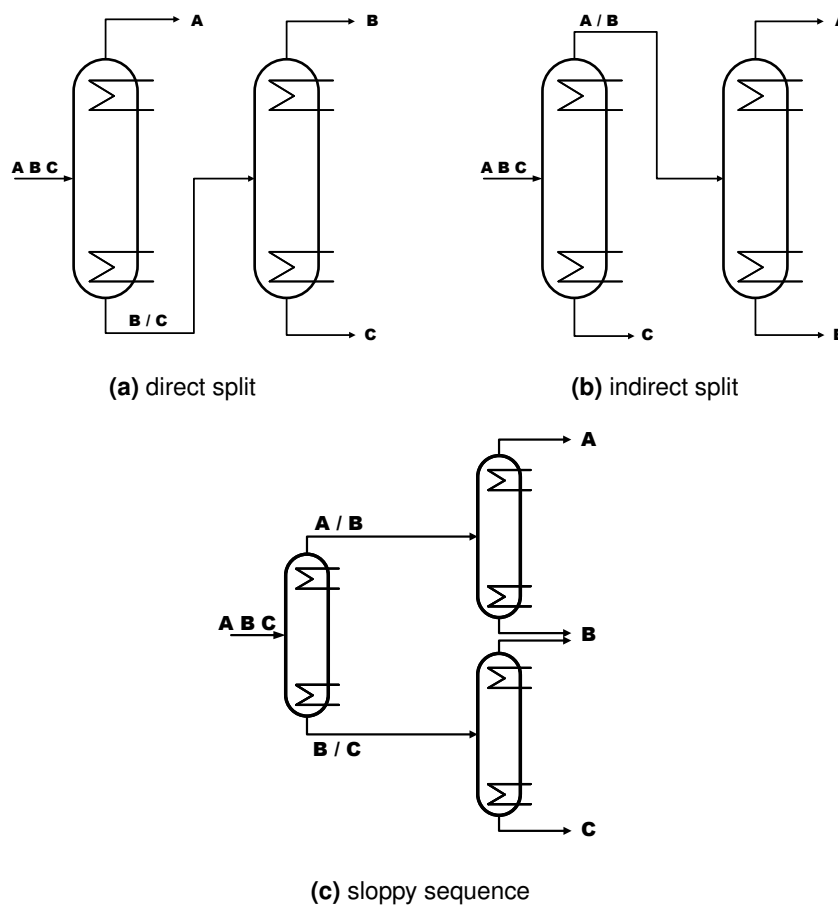


Figure 2.6: Three possible paths for separation of ternary mixture [ASPRION & KAIBEL 2010]

case B is the over head product. [RÄNGER 2021]

The sloppy sequence shown in figure 2.6c has a more preferable efficiency than the direct and indirect split configuration. The reason for this are reduced mixing losses. [DWIVEDI 2013] Nevertheless, this configuration is used less frequently because the investment costs are much higher due to the third column. [RÄNGER 2021]

The above configurations represent only a small selection of the possible options for separating ternary mixtures. If there are several components in the mixture to be separated, the possible configurations of the column sequences become even more numerous. [STICHLMAIR ET AL. 2021]

2.2 Thermal coupling and Dividing Wall Columns

Distillation is responsible for one of the largest shares of energy consumption in the process industry. For this reason, it is particularly desirable to operate this process more efficiently and thus more cost-effectively and sustainably. One possibility to reduce energy and investment costs of distillative separation of multi-component mixtures is thermal coupling of columns. [DWIVEDI 2013]

A distinction can be made between indirect and direct thermal coupling. In indirect coupling of columns, heat flows removed from one column are used to heat another column. By stepping the pressure between two columns, the heat from the condenser of one column with higher operating pressure can be used to operate a reboiler of a second column with lower pressure. [DWIVEDI 2013]

In direct or fully coupled columns material streams of several columns are connected. This allows heat flows to be used for more than one separation and subsequently increases efficiency. [DWIVEDI 2013] Direct thermal coupling will be explained using the sequence from the figure 2.6c. The direct thermal coupling of this arrangement results in the so called Petlyuk sequence.

2.2.1 Petlyuk sequence

Considering the configuration of figure 2.6c it can be seen that the subsequent columns performing the splits of AB and BC can be merged to one column. Additionally the reboiler and the condenser of the prefractionator can be replaced by a direct thermal coupling of both columns. The resulting column configuration is illustrated in figure 2.7.

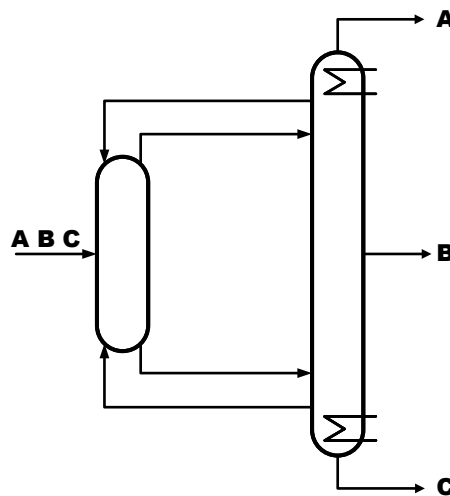


Figure 2.7: Thermally coupled columns for separation of ternary mixture proposed by PETLYUK ET AL. 1965

The resulting column arrangement is the so called Petlyuk sequence or Petlyuk column and was proposed by PETLYUK ET AL. 1965. In their work PETLYUK ET AL. 1965 showed that the thermally coupled configuration can reduce the necessary heat input required for the separation of a ternary mixture compared to the direct or indirect split. The Petlyuk column is able to save 20% to 50% of energy depending on feed composition, product specification and relative volatility. [STRANDBERG 2011]

Additionally, the investment costs are reduced due to the retrenchment of the reboiler and the condenser of the prefractionator.

One disadvantage of the Petlyuk sequence is that only one pressure level can be applied. In addition, the complexity increases because the theoretical stage number must be determined for two more column sections. [RÄNGER 2021]

Despite the reduction in energy consumption due to the sequence, two columns are still necessary. By integrating the columns into one shell with an internal separating wall, the number of columns can be reduced to a minimum. This results in a so-called dividing wall column. An illustration of this arrangement is given in figure 2.8.

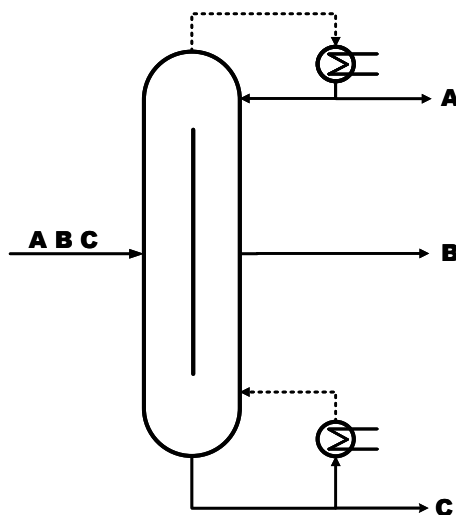


Figure 2.8: Thermally coupled Petlyuk dividing wall column for separation of ternary mixture

A Petlyuk sequence and the corresponding dividing wall column, illustrated in figure 2.7 and 2.8 are thermodynamically equivalent. [DEJANOVIĆ ET AL. 2010]

The side of the feed inlet of the dividing wall column is equivalent to the prefractionator of the Petlyuk. In this part the split between A and C is performed. The other part is equivalent to the main column where the A/B and B/C splits are performed. [STRANDBERG 2011]

Besides the reduction of energy consumption the implementation of a dividing wall column causes lower investment costs than the implementation of an equivalent Petlyuk sequence using several columns. [STRANDBERG 2011]

The principle of the Petlyuk sequence can be extended as well for mixtures with more than three components. For this, more than two columns must be implemented in the Petlyuk sequence. In the case of dividing wall column, additional dividing walls must be inserted into the column accordingly. The number of the dividing walls depends on the number of compounds in the feed mixture. [RÄNGER 2021]

For illustration, figure 2.9 shows a schematic of four-product Petlyuk dividing wall column. The illustrated column has three dividing walls.

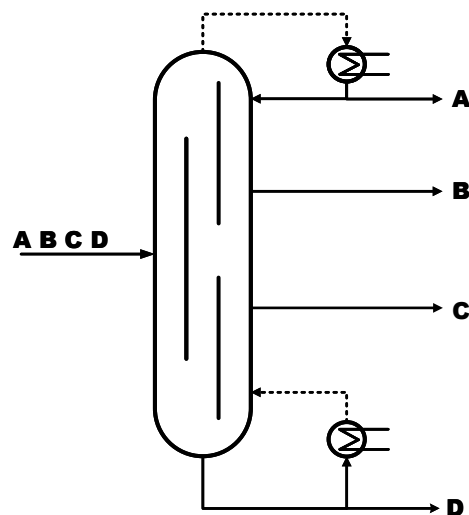


Figure 2.9: Schematic illustration of a four-product Petlyuk dividing wall column

2.2.2 Kaibel column

The paper of KAIBEL 1987 tended attention to the concept of dividing wall columns and its development potential. In addition KAIBEL 1987 suggested another kind of dividing wall column to separate four-component mixtures in his paper. This column configuration is called the Kaibel column as tribute to its inventor. A schematic illustration of the Kaibel column is given in figure 2.10.

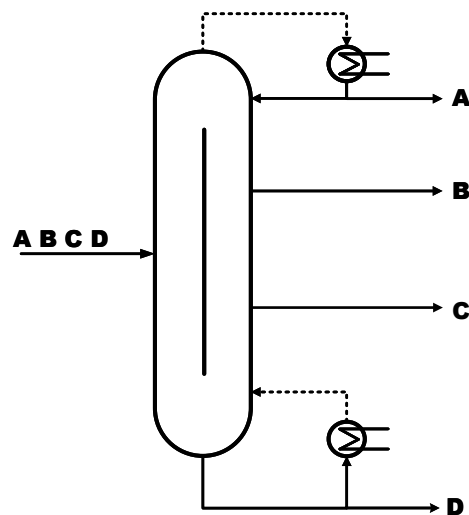


Figure 2.10: Schematic illustration of a dividing wall column to separate a four-component mixture proposed by KAIBEL 1987

The Kaibel column has only one internal partition and is capable of separating a four component mixture. Accordingly, this configuration represents a possibility to replace a sequence of three columns, which is shown in figure 2.11.

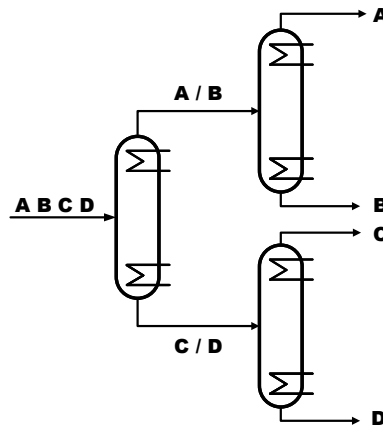


Figure 2.11: Sequence of three columns which can be replaced by a Kaibel column

This corresponds to a large potential for saving investment costs since only one column with only one dividing wall is needed. Further the Kaibel column has a large potential of reduction of energy consumption compared to a classic sequence of columns. The use of a Kaibel column can save up to 30%. [STRANDBERG 2011; HALVORSEN & SKOGESTAD 2006]

Compared to a corresponding multiple dividing wall column according to Petlyuk's concept, a Kaibel column has always a larger vapor demand. This is equivalent with a higher energy demand. [GHADRAN 2014]

Therefore a multiple dividing wall column has to be implemented to achieve the thermodynamically optimal solution for separating a four-component mixture. A corresponding configuration is shown in figure 2.9. However, this configuration is accompanied by greater complexity in terms of design and control. For this reason, there is a lack of industrial applications of multiple dividing wall columns yet. However, the first pilot-scale multiple dividing wall column was recently put into operation at the University of Ulm. [WAIBEL ET AL. 2023]

The subject of this work is a Kaibel column. Experiments are carried out on a corresponding pilot plant.

2.3 V_{\min} Diagrams

In this section the concept and basics of the V_{\min} diagram are described. The V_{\min} diagram was developed by HALVORSEN & SKOGESTAD 2003 and is based on the equations of UNDERWOOD 1948. The V_{\min} diagram is used to estimate the minimum vapor requirement for the separation in conventional and thermally coupled distillation columns. Therefore, the V_{\min} diagram gives insight to design, operation and control of a column. [GHADRAN 2014]

A conventional binary column has two degrees of freedom. Therefore the complete process can be described by a two-dimensional plot of the feed-related vapor stream V/F and the feed-related distillate stream D/F . Any combination of these two variables are

possible. Therefore it is more meaningful to identify the minimum vapor stream that is necessary to achieve a certain product purity. [RÄNGER ET AL. 2018]

The figure 2.12 shows a exemplary V_{\min} diagram of a ternary mixture in a binary column with infinite stages and liquid feed.

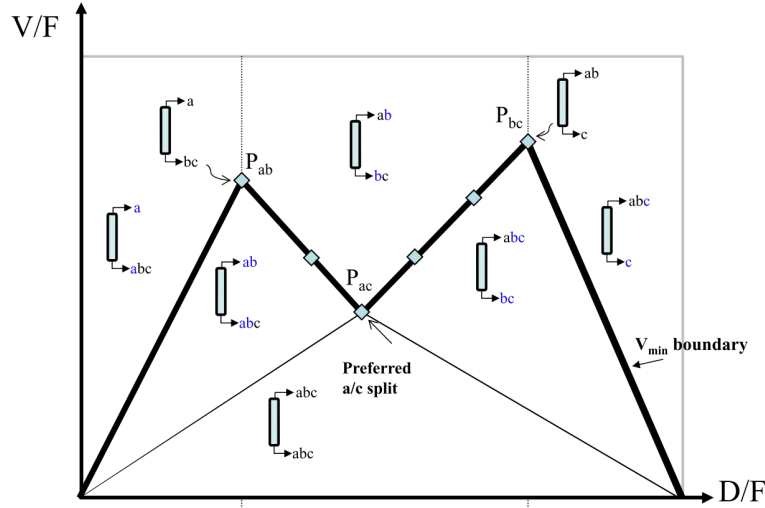


Figure 2.12: V_{\min} diagram for a ternary mixture in a conventional binary column [GHADRAN 2014]

The V_{\min} diagram in figure 2.12 shows how the components of the ternary mixture can be gained as bottom or top products with different distributions. The fat solid line describes V/F for pure products. [GHADRAN 2014]

For values of V/F above this boundary more energy is used than necessary to obtain pure products. This is a waste of energy. If less vapor is used than indicated by the boundary, pure product can not be obtained. The two "peaks" of the boundary indicate the necessary vapor stream to perform a sharp split between neighbor components A/B and B/C. The minimum of the V_{\min} boundary in figure 2.12 indicate the so called "preferred split" of the prefractionator for a ternary mixture. For this vapor stream a sharp split between the key components A and C is possible. The intermediate boiling component B is distributed on over head and bottom product stream. Accordingly, a column operates at the preferred split when the heaviest and lightest components are sharply separated with minimum vapor flow [RÄNGER ET AL. 2018, GHADRAN 2014] In general, there are $N(N - 1)/2$ peaks and minima in a V_{\min} diagram for a mixture with N components. There are $(N - 1)$ peaks for the case with no intermediates (e.g. A/B, B/C) and there are $(N - 2)$ minima for cases with one intermediate (A/C). [GHADRAN 2014]

V_{\min} diagrams can be calculated using the Underwood equations, as already mentioned. For this purpose, some assumptions are made. These are constant molar flows, constant relative volatilities and an infinite stage number. Furthermore only feed properties have to be known for the calculation of the V_{\min} diagrams. These properties are molar fraction of all components z_i , their relative volatilities α_i and the liquid fraction of the feed q . [RÄNGER 2021]

A more detailed description of the calculation method of V_{\min} diagrams is given by RÄNGER ET AL. 2018.

The concept of V_{\min} diagrams can also be applied to systems with more than three components and to sequences of columns as well as dividing wall columns. The V_{\min} diagram for a Petlyuk configuration and a Kaibel column for the separation of a four-component mixture will be explained below.

In a Petlyuk arrangement for a four component mixture every column performs the preferred split. The columns thus separate the lightest and the heaviest components respectively at minimum vapor flow. The figure 2.13 shows a four component Petlyuk arrangement on the left and the corresponding V_{\min} diagram on the right.

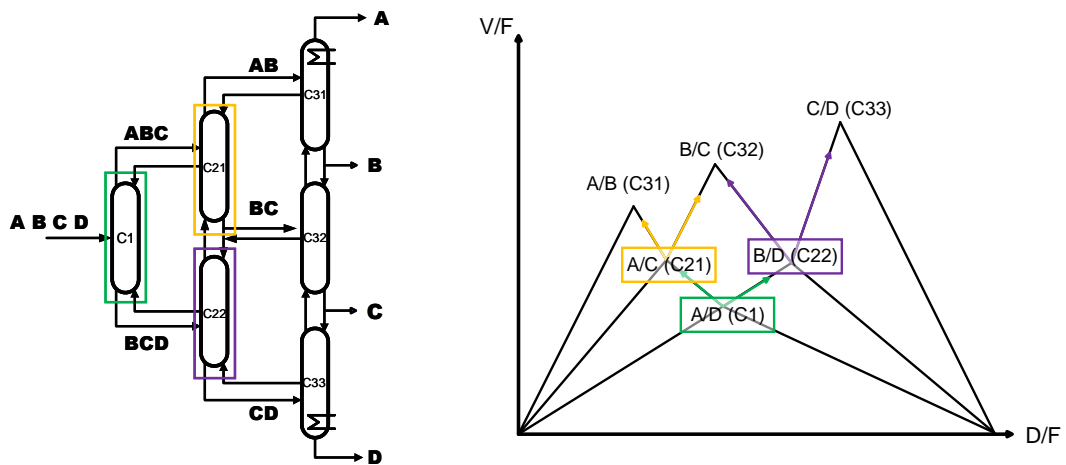


Figure 2.13: V_{\min} diagram for separation of a four component mixture in a Petlyuk arrangement

The prefractionator C1 separates the components A and D. This split is the lowest V_{\min} point in the diagram 2.13. The subsequent column C21 performs the split between A and C. The column connected to the bottom stream of the prefractionator C22 splits the components B and D. The following three columns each perform the sharp separation without distributing components. [RÄNGER ET AL. 2018]

The V_{\min} diagram will stay the same as long as every column works at the respective preferred split. The highest V_{\min} peak in the diagram defines the necessary vapor demand for the separation of the Petlyuk arrangement. Every vapor and liquid stream of a Petlyuk arrangement can be determined with the corresponding V_{\min} diagram by a balance around a certain column. [RÄNGER ET AL. 2018]

The prefractionator of a Kaibel column is not operated at the preferred split. Instead the prefractionator performs a energetically less efficient sharp B/C split. The subsequent main column performs the A/B split and the C/D split. The diagram of the original feed is only valid for the prefractionator. The minimum vapor demand for the subsequent columns has to be calculated again. This results in the V_{\min} diagram in figure 2.14. A energetically equivalent column sequence to the Kaibel column is shown on the left side of figure 2.14.

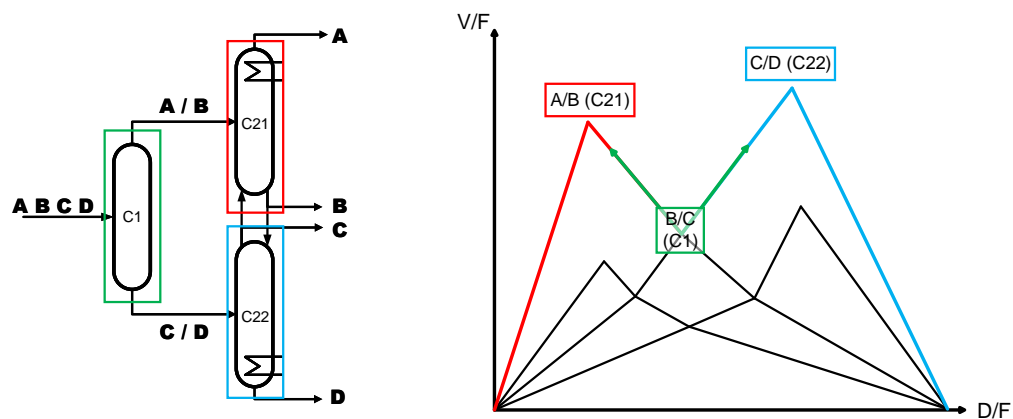


Figure 2.14: V_{\min} diagram for separation of a four component mixture in a Kaibel column

The vapor flow in the subsequent columns always need to be higher than in the pre-sequent. Therefore, it is not possible to operate column C21 with a lower vapor flow than in column C1. Because of this, it is necessary that the V_{\min} diagram changes. [RÄNGER ET AL. 2018]

The minimum vapor flow requirement for the Kaibel column is determined by the highest vapor demand for the A/B or the C/D split. [GHADRDAN 2014]

2.4 Control of Dividing Wall and Kaibel Columns

The main reason for the slow implementation of dividing wall columns in industrial applications is the challenging control of these columns. The complex control structure and interactions among different control loops resulted in a fear of problems in operation of dividing wall columns. Several researches have investigated the controllability of dividing wall columns.

DWIVEDI ET AL. 2013 propose four different composition control schemes to handle varying feed composition disturbances in a three product Petlyuk arrangement. Two of these schemes use the vapor split as a degree of freedom. A controllable vapor split has not been implemented in industrial columns due to the complexity of the equipment yet. Therefore DWIVEDI ET AL. 2013 propose two control schemes with fixed vapor split as well. DWIVEDI ET AL. 2013 found that control schemes with fixed vapor split generally perform worse than those with controlled vapor flow.

In the paper of QIAN ET AL. 2016a three temperature control schemes for a three product dividing wall column are compared. In the paper a control structure with fixed split ratios, a structure with active liquid split, and a control structure with active vapor splits are considered. QIAN ET AL. 2016a have found that all three structures are able to handle the feed disturbances. A vapor split disturbance is better handled by the structures with active vapor split control. However, the simple control structure with fixed ratios is considered as more applicable in industry. QIAN ET AL. 2016a have proven that a three-product dividing wall column could be controlled by a three-point

temperature control.

STRANDBERG & SKOGESTAD 2006 investigated the controllability of a four-product Kaibel column. Using a simulation study, they were able to show that a Kaibel column can be stabilized with a four-point temperature control structure. Thereby one temperature in the prefractionator is controlled. Furthermore the investigated control structure was able to prevent a drift of the composition profiles during operation. The figure 2.15 shows the considered four-point control structure.

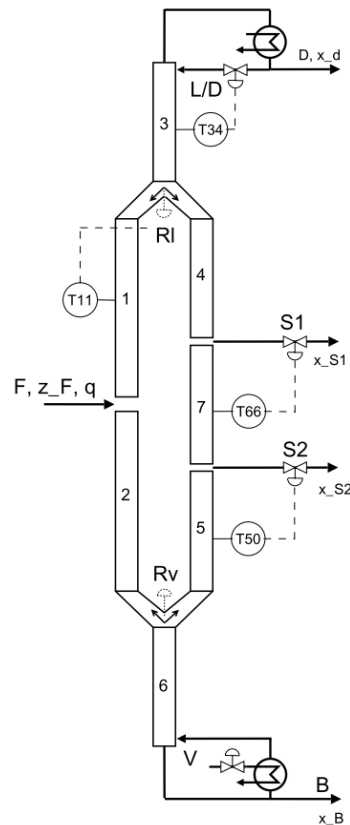


Figure 2.15: Four-point temperature control structure to stabilize a Kaibel column from the paper of STRANDBERG & SKOGESTAD 2006

From the figure 2.15 it can be seen that the reflux ratio L/D , the two flows of the side products and the liquid split R_L are controlled.

In their paper, QIAN ET AL. 2016b presented three other structures for controlling a four-product Kaibel column. The first control structure uses only temperature controllers, while the other two additionally use composition controllers. The authors could show that the control structure which only uses temperature controllers is able to handle feed disturbances surprisingly well. Additionally the simple temperature control structure showed a better dynamic performance with smaller dynamic deviations than the structures with composition control.

2.4.1 Active Vapor Split

Compared to conventional columns, dividing wall columns have two additional degrees of freedom. These degrees of freedom are the vapor and the liquid split at the bottom and the top of the dividing wall. Especially a correct adjustment of the vapor split is necessary to operate a dividing wall column at the energetically most favorable operating point. Accordingly, an active vapor split control is necessary to ensure flexibility during operation with minimal energy requirements even with fluctuating feed parameters and product specifications.

As already mentioned above, the implementation of an active vapor split is challenging and therefore no industrial applications are known so far.

STRANDBERG 2011 describes in his doctoral thesis the construction of valves for active vapor split in a lab sized Kaibel column. The same column is used in the present thesis. At first, STRANDBERG 2011 installed simple butterfly valves in the column, which were operated manually and were only poorly able to control the vapor flow. Because of this, different valves were constructed which used a rack and pinion arrangement. First experiments of STRANDBERG 2011 showed that the valves were able to manipulate the vapor flow inside the column to some extent. The design of the valves is still too large, this leads to the fact that 95% of the opening range corresponds to a fully opened valve and thus has no influence on the vapor flow.

In the paper of DWIVEDI ET AL. 2012 the same lab column and vapor split valves are used. The authors investigated the controllability of a dividing wall column using an active vapor split. A four-point temperature control with a constant liquid split was used. In the experiments, the side product streams, the distillate stream, and the vapor split were controlled. DWIVEDI ET AL. 2012 could show that the vapor split can be used as a degree of freedom, for temperature control in the prefractionator section. Although there is great potential for improvement in terms of vapor valve design, it has been shown experimentally that the use of the vapor split in feedback loop allows for more energy efficient operation. DWIVEDI ET AL. 2012 also cite that the vapor split is a fast handle compared to the liquid split since the vapor dynamics are much faster.

In his doctoral thesis DWIVEDI 2013 investigates the control of a Kaibel dividing wall column using the active vapor split control. The same experimental setup is used as by DWIVEDI ET AL. 2012. The thesis by DWIVEDI 2013 is a comprehensive study on the impact of using active vapor split in different dividing wall columns on the energy consumption. It is shown that there is a close link between energy consumption and vapor split and that a suboptimal vapor split leads to increased energy consumption.

GE ET AL. 2014 presented a different design for a vapor split valve in their experimental study. In the proposed design, the vapor flow is manipulated by adjustable blades. These blades are located in a vapor channel and are adjusted through axles by an external motor. The liquid passes the valve through a downcomer. The authors conclude that the valve can adjust the vapor flow regardless of the operating conditions. However, the design still has some limitations.

In their experimental study, KANG ET AL. 2017 introduced a new kind of active vapor distributor. The proposed vapor splitter is a hydraulic driven device. The splitter

consists of two valves for liquid level control and several vapor chimneys with windows as vapor channels. The vapor flow is controlled by adjusting the liquid level in each section. The liquid flow from upper to lower trays and thus the liquid level is adjusted by control valves. The advantage of the proposed design is that it lacks any moving or mechanical parts like motors or moving blades. Therefore the investment costs and the susceptibility of the design to failure are low. The authors could show that the proposed active vapor splitter is able to split the vapor flow during operation.

3 Experimental setup and procedure

In this chapter the experimental setup of the Kaibel column pilot plant is described. Furthermore the design of the vapor split valves and the control structure is shown. The control software and the improvements made to the software during the thesis are explained in the following section. At the end of the chapter a description of the experimental procedure is given.

3.1 Kaibel column

The investigated Kaibel column is located in laboratory hall C at the Department of Chemical Engineering of NTNU in Trondheim. The pilot plant was built during the Ph.D. studies by STRANDBERG 2011. The column is realized as a two branch design which is thermodynamically equivalent to a dividing wall column design. The figure 3.1 shows a picture of the column as well as schematic illustrations of the positions of temperature sensors and the used control structure.

The Kaibel column consists of several glass parts which are double-walled. The inside of the double wall is evacuated and the inside is silver-coated to minimize heat loss. All glass sections were produced by *Normag Labortechnik* in Germany. The single glass sections are connected with flanges and mounted with screws. To avoid stresses in the structure, the entire column is flexibly suspended on springs at the connecting flanges. Every glass section has a inner diameter of 50 mm except the sections directly above the reboiler which has a inner diameter of 70 mm. The height of the lab column is about 8 m.

The single sections are numbered from 1 to 7, as can be seen in figure 3.1 to the right. The sections 1 and 2 constitute the prefractionator of the Kaibel column, whereas sections 3 to 7 are equivalent to the main column.

The Kaibel column is packed with a random packing of glass Raschig Rings. The Raschig Rings have a diameter of 6 mm. The packed beds in each section rest on a short piece of structured packing. The height of the packings in section 1 and 2 are 1.1 m and 1.6 m. While the packed beds in sections 3, 4 and 5 are 0.65 m. The random packed beds in sections 6 and 7 are about 0.75 m and 0.9 m. [DWIVEDI 2013]

The heat for the lab column is provided by a kettle-type reboiler at the bottom of the column. This electric reboiler can provide a maximal heat input of 2.9 kW. The power to the reboiler is adjusted by varying the voltage through a thyristor. The maximum capacity of the reboiler is 15 L and the operation minimum is 3 L.

The liquid reflux is ensured by a water-cooled condenser at the top of the column. The product streams at the side of the column are water-cooled as well to ensure low

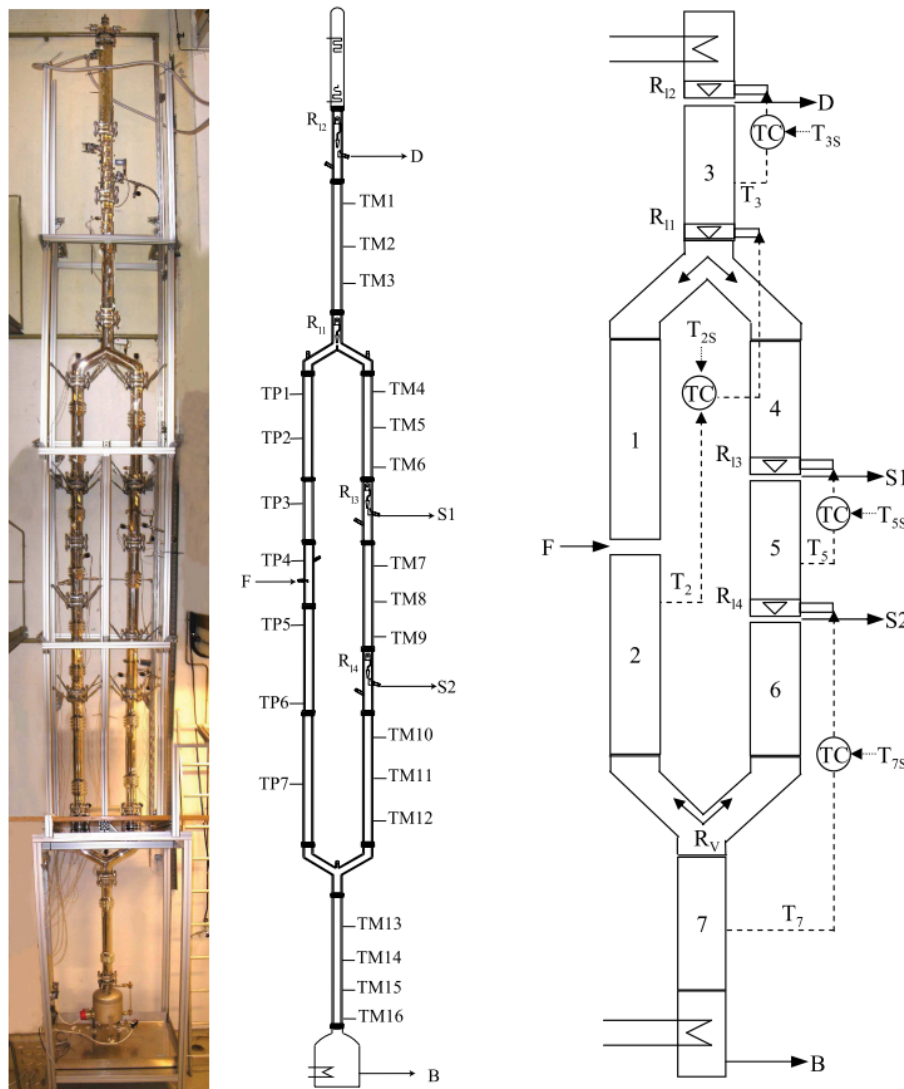


Figure 3.1: Picture of the investigated Kaibel column (left), Locations of temperature sensors (center), Schematic of the 4-point control structure right, [DWIVEDI 2013]

temperatures of the products. Therefore small heat exchangers are installed at the side streams.

The valves of the liquid split and the product withdrawals are designed as swinging funnels. These funnels are controlled (On/Off) by external solenoids. The valve of the liquid split directs the liquid flow either to the prefractionator or to the main column. The valves at the product outlets carry out a fraction of the liquid flow in the column as product flow.

Since the swinging funnels are operating at On/Off states only, the common technique of pulse width modulation (PWM) is used to emulate continuous flow conditions. The width and period using pulse width modulation is normally fixed. In our case, the cycle time of the valve switching function is about 10 s and the resolution is 0.2 s. For example, if the controller output for a valve is 0.44, the funnel position is for 4.4 s on one side and for 5.6 s on the other. To improve the resolution DWIVEDI 2013 implemented an

algorithm which allows to change the cycle time between 5 s and 15 s. This improvement reduces rounding off errors.

The feed is conducted to the column with a digital diaphragm dosing pump of the type *TrueDos* manufactured by *Alldos*. The pump is controlled through the control software of the rig. The pump is able to provide a feed flow with a rate from 0.2 L/h to 20 L/h. A heating band is installed around the feed pipe to preheat the feed flow before entering the column.

The Kaibel column is operated at atmospheric pressure and is therefore open to the environment. A sufficient cooling power of the condenser prevents escaping vapors. Due to the vaporization in the reboiler and the pressure drop of the packing, a certain over pressure is created in the column. To prevent vapor escaping at the product outlets, liquid seals are installed. These seals consist of U-tubes filled with liquid.

In case of critical failures of the column it is necessary to shut down the system as quickly as possible. Critical failures are leakages or interrupted cooling water supply for example. For this case two emergency buttons are installed on 1st and 2nd floor. By pressing one of these buttons the power supply to the column is immediately disconnected.

3.1.1 Vapor split valves

It is possible to manipulate the vapor split of the investigated Kaibel column at the lower branching using vapor split valves. The vapor split valves were developed by STRANDBERG 2011. The purpose of the valves is to control the split of vapor flow between the prefractionator and the main column to achieve a more energy efficient operation. The figure 3.2 shows a schematic illustration of the design of the vapor split valves.

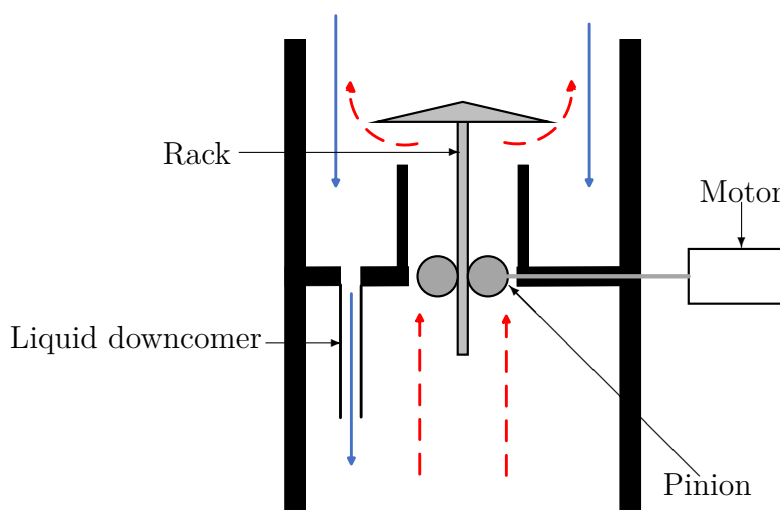


Figure 3.2: Schematic illustration of the design of a vapor split valve

The valve is designed to fit as a section between two glass sections of the Kaibel column. The vapor flow is manipulated by using a rack and pinion arrangement. A cap is mounted on top of the rack. The vapor flow can be adjusted by opening or closing the cap via an external stepper motor. By closing the cap, the diameter is reduced and therefore the flow is reduced as well. This principle can be seen in figure 3.2.

The liquid which flows from the upper column parts is collected by a tray. A liquid downcomer is installed so that the liquid can pass the valve. The bottom end of the downcomer is constricted so that a certain liquid level on the tray is ensured. This forms a liquid barrier to the vapor.

The two vapor valves are located at the lower branching of the Kaibel column. They are installed at the junctions between section 2 and 7 before the prefractionator and between section 6 and 7 before the main column, respectively.

The current design of the valves still offers potential for improvement. The opening of the valves are too large, so that the majority of the opening range has no influence on the vapor flow and the vapor split. Only about the last 10 steps of the stepper motor affect the vapor flow as shown by DWIVEDI 2013. Nevertheless, the valves are able to adjust the vapor flow.

The vapor split valves are used to implement a active vapor split control to achieve an energy effective operation of the Kaibel column. Therefore the temperature $TP7$ in the prefractionator, which is shown in the center of figure 3.1 is used as controlled variable. The ratio between vapor flow to the prefractionator and the total flow from reboiler is used as manipulated variable.

A picture of a dismantled vapor split valve can be seen in figure 3.3.



Figure 3.3: Picture of dismantled vapor split valve, [DWIVEDI 2013]

3.1.2 Control structure

The control structure is implemented in the control software in *LabView*, which is explained in a following section explicitly. Up to five feedback loops can be implemented

in the structure to ensure stabilized operation. The measured temperatures in the column are used as controlled variables.

As shown by STRANDBERG & SKOGESTAD 2006 a four-point temperature control is able to stabilize the operation of a four-product Kaibel column and prevent drift of composition. In figure 3.1 the "standard" four-point control structure with manipulation of the liquid split R_L is shown as an example.

Different degrees of freedom of the lab column can be chosen as manipulated variables and therefore as controller outputs. The feed rate and composition as well as the reboiler duty were kept constant and not used as manipulated variables. The other degrees of freedom were used as manipulated variables in the control structure. These controller outputs u are summarized in the following list:

- Distillate product split u_D , ratio between liquid flow from the top and total liquid flow of condensate:

$$u_D = \frac{L_3}{L_3 + D} \quad (3.1)$$

- Upper side product split u_{S1} , ratio between liquid flow below the side stream 1 withdrawal and liquid flow above the side stream 1 withdrawal:

$$u_{S1} = \frac{L_5}{L_5 + S_1} \quad (3.2)$$

- Lower side product split u_{S2} , ratio between liquid flow below the side stream 2 withdrawal and liquid flow above the side stream 2 withdrawal:

$$u_{S2} = \frac{L_6}{L_6 + S_2} \quad (3.3)$$

- Liquid split R_L , ratio between liquid flow to prefractionator and total liquid flow from section above:

$$R_L = \frac{L_1}{L_3} \quad (3.4)$$

- Vapor split R_V , ratio between vapor flow to prefractionator and total vapor flow from reboiler:

$$R_V = \frac{V_2}{V_7} \quad (3.5)$$

Hereby L denotes liquid flows and V denotes vapor flows inside different sections of the column. The notations are equivalent to those in figure 3.1 on the right.

During the experimental work, different control structures were used. In the "standard" case, the control loops of the three or two product withdrawals were used together with the control of the liquid split. In addition, instead of the liquid split, the vapor split

can be controlled in conjunction with the product streams. A five-point control scheme with both liquid and vapor split control is also possible. This was investigated by KORBELAROVA 2018 in her Master's Thesis. In case the column is only operated with a three-component mixture, the product side stream S_1 is not controlled but closed. The used control loops are summarized in table 3.1 together with the controlled temperatures and manipulated variables.

Table 3.1: Feedback control loops with corresponding variables

control loop	manipulated variable	controlled temperature
distillate stream	D	TM2
upper side stream	S_1	TM8
lower side stream	S_2	TM11
liquid split	R_L	TP2
vapor split	R_V	TP7

In general, the control loops are implemented in the software in such a way that the controlled temperatures for each loop can be freely selected and adjusted.

In addition to the mentioned control loops, there is another loop for preheating the feed stream. This controller adjusts the temperature of the heating band on the feed pipe. The preheating of the feed uses the same logic as the swinging funnels of the split and product valves, since the heating band can only take the states On and Off. Hereby the pulse width modulation is used as well to control the preheating of the feed. The preheating control loop uses an inverted logic since the Off time is used instead of the On time for PWM.

The feed flow and the reboiler duty are set by an analogous voltage output which are calibrated to fit the respective units. These variables are not used as automatic controller outputs.

3.2 Control Software in *LabView*

The control interface of the lab Kaibel column is implemented as a Virtual Instrument (VI) in *LabView*. *LabView* is a development environment for a visual programming language sold by the company *National Instruments*. It is often used in measurement, control and automation technology.

The purpose of the control software is to monitor and control the Kaibel column during operation. In addition, the software is used to record the measurement data of the sensors and the controller settings. The software was programmed during the Ph.D. studies of STRANDBERG 2011 along the construction of the lab column. Over time, further improvements and functionalities were added. A Screenshot of the software interface, the so-called Front Panel is shown in figure 3.4.

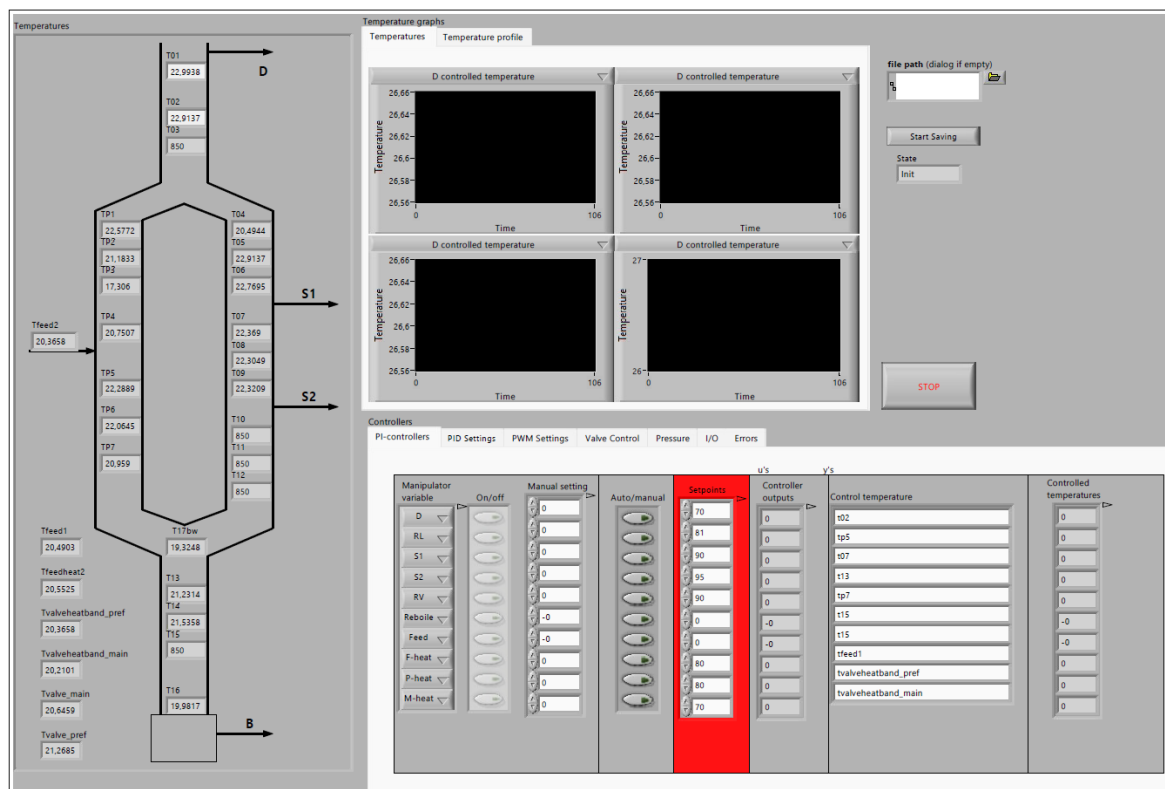


Figure 3.4: Screenshot of the control software interface in LabView

At the left side of figure 3.4 a schematic chart of the column is observable. This chart shows the currently measured temperature values at each position along the column. The measurement values are updated every measurement cycle which is every second. Four charts show the profile of eligible temperatures for the last 100 measurement points in the upper part of the interface. With the help of these diagrams, the development of the temperature curves during operation can be observed and controlled. At the same position the temperature profiles of the prefractionator and the main column can be shown by selecting the corresponding chart from a task bar.

A text box is located right next to these four charts. A file path as well as a file name can be entered in this box to record the measurement data. The file format is a simple text file. By clicking the button "Start Saving" the data recording is started. The resulting file can then be opened and evaluated in Excel or MATLAB.

Different functions are implemented in the task menu at the lower part of the interface. The most important function is shown in the screenshot in figure 3.4. The settings of controllers are defined in the shown chart. At the left side the manipulated variables can be selected and the controllers can be activated with binary switches. The middle button is used to select between manual or auto mode. The setpoints of the controllers are defined in the red text box of the chart. The outputs of the controllers are indicated next to the red text box, respectively. At the right side of the chart, the controlled temperatures can be selected and the current temperature values are displayed.

In the other tabs of the Task menu, further settings for the controllers can be made. In addition, the vapor split valves can be controlled manually in a separate tab.

The complete structure and functionality of the control software is not described here, as this would go beyond the scope of this paper.

3.2.1 Implemented changes and improvements to the software

One of the main tasks of the research internship, in the course of which this paper was written, was to update and improve the control software. The version of *LabView* running the control software as well as the operating system of the PC should be updated. To this point *LabView 2009* on *Windows Vista* was used. Since this software version and operating system are almost 15 years old, updating was necessary. This task was quite difficult and time consuming. Extensive familiarization and research on this subject area was necessary.

Transferring *LabView* codes from older versions to newer versions is often difficult. The reason for this is that functionalities are removed, changed or replaced by new functionalities during the release process. This often makes it necessary to adapt the code, which is not always simply possible.

As new operating system, *Windows 10* was chosen and installed on a new PC in the lab. When trying to update the version of *LabView*, a problem appeared. The *LabView* Code uses the hardware and software system FieldPoint IO to communicate with the sensors of the column. The support of FieldPoint was discontinued by *National Instruments* after *LabView 2013*. Since it was not planned to replace any hardware, the latest version to update to is *LabView 2013*.

LabView 2013 was installed on the PC. After all necessary settings for the communication with the sensors and actuators of the column were made, the column could be put into operation using the updated control software. At this point, not all necessary changes and settings are mentioned, as this would exceed the scope of this work.

Furthermore a safety switch off was implemented in the control software. One of the main risks during operation of the column is the ignition of chemicals due to overheating of the reboiler. This may happen if the liquid level inside the reboiler is insufficient. If the level is too low, the temperature at the reboiler rises above the boiling temperature of the heavy component because the vapor is overheated. This temperature is measured by the temperature sensor *TM16*.

The implemented code compares the measured temperature value with a certain limit value during every measurement cycle. If the measured value exceeds the limit, the implemented safety switch off is triggered. The code section representing the safety switch off is shown in figure 3.5.

As can be seen in figure 3.5 the implemented code compares the measured temperature value with a fixed limit value. If the measured temperature is higher than the limit, the boolean value *true* is submitted to the stop function of the code. This makes sure that the control software code and the heat supply to the reboiler is stopped. In consequence, the column will cool down and a possible ignition of the chemicals is prevented.

In addition a message will pop up if the safety switch off is triggered to inform the user about a high temperature at the reboiler. The whole code of the safety switch off is

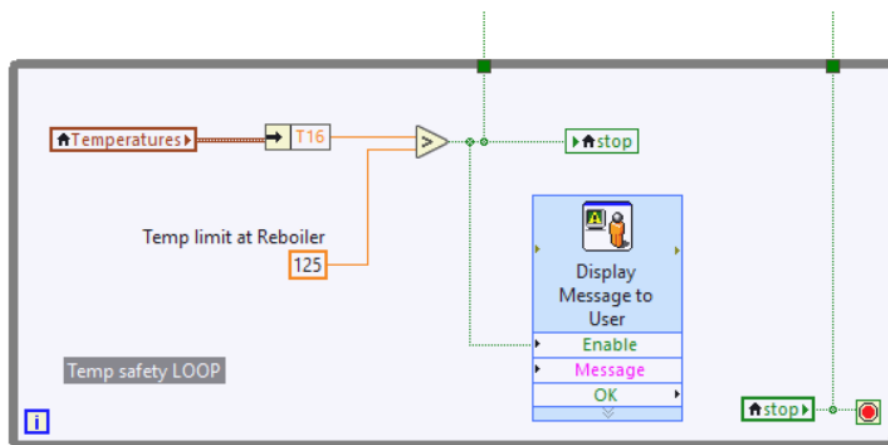


Figure 3.5: Screenshot of Block Diagram of the implemented safety switch off in *LabView*

embedded in a while-loop, as shown in figure 3.5. This safety switch off was tested by setting the limit value lower than ambient temperature and starting the software. Consequently, the program and the heat input were stopped immediately. Furthermore, the display of the currently controlled temperatures was added to the Front Panel of the control software. This can be seen in figure 3.4. This is merely a small visual improvement of the interface.

3.3 Experimental procedure

The experimental and operational procedure of the Kaibel column are described in this section. The start-up and the shut-down procedure are explained in particular. Therefore a step-by-step description of the operation process is given. In addition the used chemicals and the settings for the controllers are summarized.

A three-component mixture of ethanol, 1-propanol and 1-butanol were investigated. Although the column was designed for a four-component mixture, experiments were conducted with only three components. This was because the use of methanol as a fourth light boiling component was avoided due to health hazards of this alcohol.

Two different compositions of these components were investigated. At first a mixture consisting of $x_{\text{ethanol}} = 0.15$, $x_{\text{propanol}} = 0.55$ and $x_{\text{butanol}} = 0.3$ was used. In later experiments a mixture of $x_{\text{ethanol}} = 0.25$, $x_{\text{propanol}} = 0.45$ and $x_{\text{butanol}} = 0.3$ was used to increase the flow of the top product.

Since only a three-component mixture was used, the S_1 product side draw was closed and only the S_2 outlet was used during most of the experiments.

For the implementation of the controllers several values for the controller constants and the maximal and minimal values for the manipulated variables are needed. The used controller settings were proposed by KORBELAROVA 2018. The table 3.2 contains the controller settings.

Table 3.2: Controller settings for used feedback loops proposed by KORBELAROVA 2018

manipulated variable	Min-Max	Kp	I	controlled variable
D	0-1	-0.3	4	TM2
S_1	0-1	-0.18	4	TM8
S_2	0-1	-0.32	4.16	TM13/TM11
R_L	0-1	-0.03	4	TP5/TP2
R_V	0.05-0.95	0.09	8	TP7
Feed preheat	1-0	-0.03	10	Feedheat1
Feed flow	0-10	-	-	-
Reboiler duty	0-2	-	-	-

Since a different feed mixture was used in this work, the setpoints proposed by KORBELAROVA 2018 could not be used. It is necessary to find alternative setpoints for the control loops.

3.3.1 Column start-up

In this section the procedure to start-up the Kaibel column is explained. The description is structured as a step-by-step guide.

Before starting the column, the rig must be inspected for possible leakages or obvious damage. Especially the glass sections and junctions should be inspected. Leakages can also be detected by smell of alcohols.

Furthermore the liquid levels of the reboiler and the feed tank need to be checked to guarantee a safe operation of the device. As already mentioned, a insufficient liquid level in the reboiler might lead to overheating of the heating rods and to ignition of the alcohol mixture. If the reboiler level is too low, feed must be added via the feed pump. If necessary feed mixture must be added to the feed tank.

The next step is to turn on the feed pump. The pump is connected to a separate power supply. Analog mode must be selected at the pump. Before column start-up the pump must be purged via the bypass to remove any air inside the pump or the flexible pipes. Proper feed flow must be observed before start-up.

To minimize the risk of ignition of vapors inside the rig, the column is purged with nitrogen before start-up. This can be done by a direct connection from the nitrogen socket at the wall to the bottom of the column.

Before starting the heat input proper cooling water flow must be ensured. The valves controlling cooling water inlet and outlet must be fully opened to ensure sufficient cooling duty of the condenser. In addition the valve of the product outlet cooling must be opened. Hereby a proper flow without bubbles must be ensured.

In the next step the power supply can be switched on and the PC can be started. The power supply is turned on at the electrical cabinet on 2nd floor. The control software in *LabView* can be started as well. The column is controlled via the *LabView* interface.

After the start-up of the control software, the settings of the controllers can be made. The controller settings are summarized in table 3.2. Additionally, the feed flow rate and the reboiler duty can be set. The controllers should not be activated at this point. In addition, the correct position of the vapor valves must be checked, as there may be a discrepancy between the position displayed in the software and the real position of the valves. If necessary the positions of the valves can be reset via the control software. After ensuring that all settings have been made correctly and the column is ready, the reboiler can be started. This starts the heat-up of the column. At this point the recording of the measurement data should be started. During heat-up all controllers should be in manual mode. The feed input is not active during heat-up. The liquid split ratio R_L is set to $R_L = 0.5$ to achieve uniform distribution of the liquid. Furthermore all product withdrawals are closed and the vapor split valves are fully opened until the column is heated up. The column is operated at total reflux during heat-up. As soon as the temperature profile of the column is stabilized, the controllers can be activated one after the other. Additionally the feed input can be started by activating the feed pump via the control software. If applicable the preheating of the feed flow can be started as well. Once all of the temperatures are stabilized, the experiments can be conducted.

3.3.2 Column shut-down

After all experiments are conducted the rig can be shut down. The shut-down procedure of the Kaibel column is described in this section.

The first step is to stop any heat input to the column. Therefore the reboiler duty is set to zero in the control software. In addition the pre-heating of the feed flow must be stopped. After the heat input is cut off the column begins to cool down. As soon as the heaters are switched off the feed flow can be set to zero in the *LabView* interface. As a following step all controllers can be switched off. The controller outputs of the product withdraws and the liquid split are set to 0. Thereby the remaining liquid is withdrawn and flows to the product tanks. This ensures that the liquid that collects in the reboiler is mainly heavy boiling component. After this the data recording can be stopped. The measurement file is saved and a backup of the file is created.

After this is done it has to be waited until the column has cooled down. This takes about 30 minutes. The flow of cooling water to the condenser must not yet be interrupted. Otherwise vapors might escape at the top of the column.

As soon as the column is cooled down, the software and the PC can be shut down. Do not save the changes to the *LabView* if a pop-up window appears. After the PC is shut down, the power supply to the PC and the column can be cut off as well. Ensure that the power is switched off by pressing the red emergency button at the electrical cabinet. The next step is to close the valves of the cooling water at the top floor. Furthermore the feed pump at the first floor must be turned off since this device is connected to a separate power supply. The products in the tanks can be recirculated to the feed tank after the run.

4 Results and Discussion

The conducted experimental runs are described and evaluated in this section. Additionally the obtained results are discussed in this part of the paper.

The goal of the experiments was to validate the operability of the Kaibel column and the respective temperature controllers. Several experiments with different control arrangements were conducted to investigate the respective impacts of the controlled variables on the operation. Therefore the liquid and the vapor splits were controlled separately as well as simultaneously in a 4-point control scheme in several experimental runs.

Unfortunately not all planned experiments could be conducted since the time of the internship, during which this paper was done, was restricted. The modification and updating of the control software took a large amount of time at the beginning of the internship. Additionally, several issues of the lab column together with delayed delivery of spare parts caused further delay. Nevertheless, the measurements performed are described and discussed.

4.1 Liquid split

At first the experimental runs using only the liquid split are described. The vapor split was deactivated during these runs and the vapor split valves were fully opened.

Open loop as well as closed loop experiments with a 3-point control structure were conducted to examine the performance of the liquid split R_L .

4.1.1 Experimental run with different step changes of R_L

At the beginning the open loop experiment using the liquid split R_L is described. During this experimental run a reboiler duty of $Q = 1.8 \text{ kW}$ and a feed flow of $F = 2.0 \text{ L/h}$ were used. The feed was not preheated and entered the column with a temperature of about $T = 24 \text{ °C}$. For this experimental run a mixture of $x_{\text{ethanol}} = 0.15$, $x_{\text{propanol}} = 0.55$ and $x_{\text{butanol}} = 0.3$ was used.

The controller of the distillate product flow D used a setpoint of 82 °C and the controller of side product flow S_2 used a setpoint of 108 °C . The S_2 controller used the temperature T13 in the lower section of column as controlled variable. This temperature is too far from the product output S_2 , so a different temperature is used in the following experiments.

The setpoints were chosen based on observations during the initial experiments and based on an *Aspen Plus* simulation created in the Process Systems Engineering Group.

Due to deviating values of the temperature sensors and other factors, the setpoint temperatures do not fully agree with the simulation results and were adjusted based on observations.

The following figure 4.1 shows the plots of the controller outputs together with the controlled temperatures over the duration of the experiment.

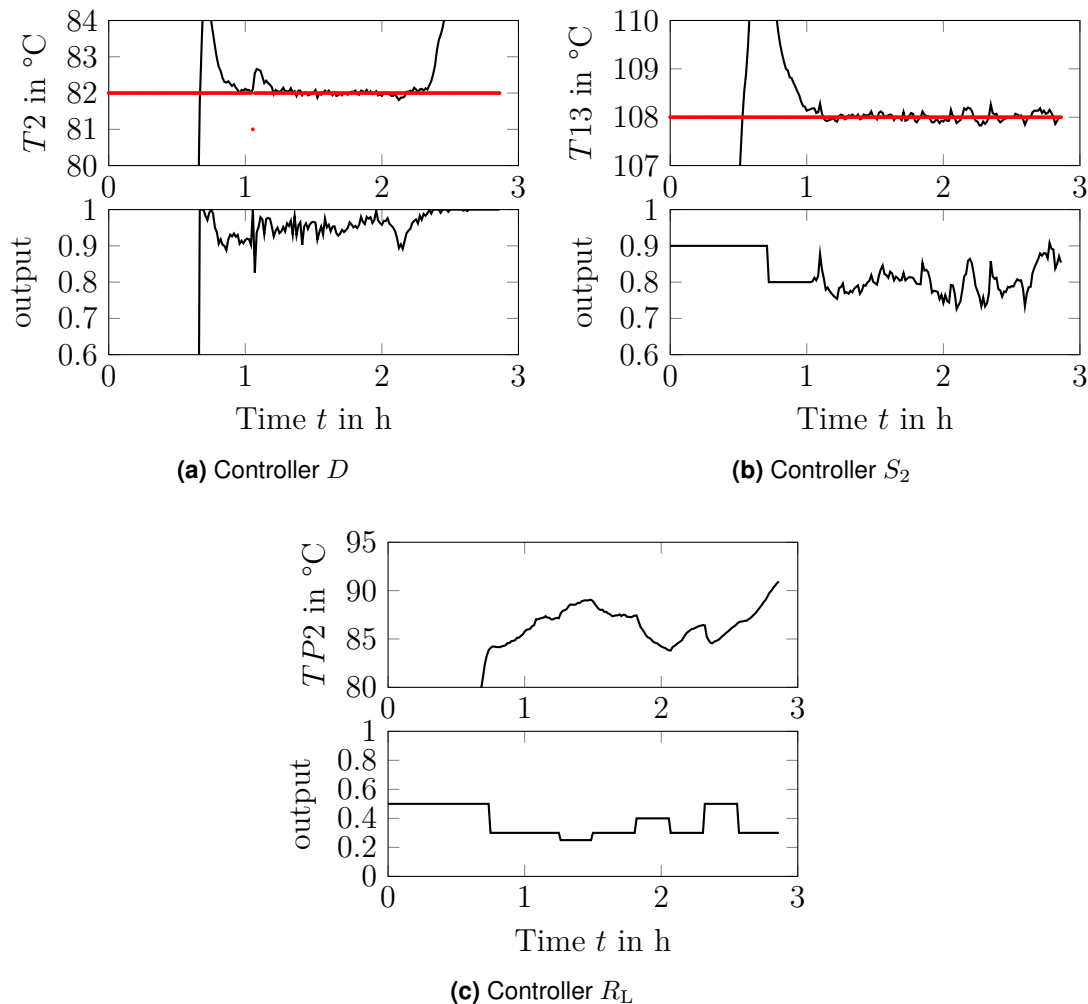


Figure 4.1: Controller outputs and controlled temperatures of D , S_2 and R_L of open loop experiment with manipulated liquid split and deactivated vapor split

As can be seen in figure 4.1 the controller D was started after 40 min as soon as the column had heated up whereas a fixed value of 0.8 was set as output of the S_2 controller. The liquid split was set from 0.5 to 0.3 at this point. This results in a higher liquid flow to the main column. The automatic control of S_2 was started after 1 h. The feed was recirculated during the experiment after about 2 h. This had no observable impact. The setpoint of controller D was reached pretty quick after about 15 min and could be obtained almost until the end the experiment. The controller S_2 achieved the temperature of the setpoint as well and was able to maintain the setpoint during the whole experiment.

During the experiment several step changes of the liquid split were introduced. The first change from 0.5 to 0.3 after the heat-up resulted in a slowly increasing temperature TP_2 ,

which can be seen in figure 4.1c. With a liquid split of 0.3 the temperature TP2 reached a constant value of 87 °C after about 1.1 h. In the next step the liquid split R_L was decreased to 0.25 after 1.25 h. This results in a higher liquid flow to the main column. Therefore the temperature TP2, which is located at the top of the prefractionator increased to 89 °C after 1.5 h. After that the liquid split was set back to 0.3 and the temperature TP2 decreased to 87 °C again. After 1.75 h the liquid split was increased to 0.4, which led to a higher flow to the prefractionator than before. Therefore the temperature TP2 decreased until the liquid split was set back to 0.3 after 2 h. After that the temperature rose again. A liquid split of 0.5 was set after 2.25 h, which resulted in a short decrease of TP2. A rise of the temperature TP2 was observed after about 2.4 h. The liquid split was set back to 0.3 after 2.5 h. The increase of TP2 continued until the end of the experiment.

None of the performed step changes of the liquid split had any discernible effect on the controlled temperatures of D or S_2 . Only the temperature TP2 was directly influenced. From this it can be concluded that TP2 is suitable as a controlled temperature of the liquid split.

At the end of the experiment after about 2.4 h an increase of temperature TP2 and T2 can be observed in figures 4.1c and 4.1a. This temperature increase continued until the end of the experiment. At first no obvious reason for this temperature rise was observed. After examination of the column, a falsely positioned product heat exchanger at the D outlet could be indentified as a possible reason for the temperature behavior at the end of the experiment. The heat exchanger was installed in such a way that vapor from the column was continuously condensed and withdrawn from the column. Therefore light component was continuously withdrawn which could cause the temperature rise at the end of the run. Additionally, the performance of the controller D was probably effected negatively by this. The heat exchanger was then mounted so that the liquid seal is located in front of the cooler to prevent uncontrolled condensation of vapor.

4.1.2 Experimental run with controlled liquid split R_L

In the next step a closed loop experiment with automatic controlled liquid split R_L was conducted. This experimental run is described in the following section. The reboiler duty was set to $Q = 1.8$ kW and the feed flow was set to $F = 2.0$ L/h. The feed was not preheated and entered the column with ambient temperature. For this experiment the mixture consisting of $x_{\text{ethanol}} = 0.25$, $x_{\text{propanol}} = 0.45$ and $x_{\text{butanol}} = 0.3$ was used. A temperature of 82 °C was chosen as setpoint for the controlled temperature T2 of controller D . In contrast to the open loop experiment of the liquid split, in this experiment the temperature T11 was chosen as the controlled variable for the controller S_2 . A temperature of 100 °C was chosen as setpoint for this controller S_2 . The liquid split control used the temperature TP2 at the top of the prefractionator as controlled variable. A temperature of 85 °C was used as setpoint for the liquid split control. The choice of setpoints was again based on observations from previous experiments.

The figure 4.2 shows the plots of the controller outputs and the trend of the controlled temperatures of the controllers D , S_2 and R_L .

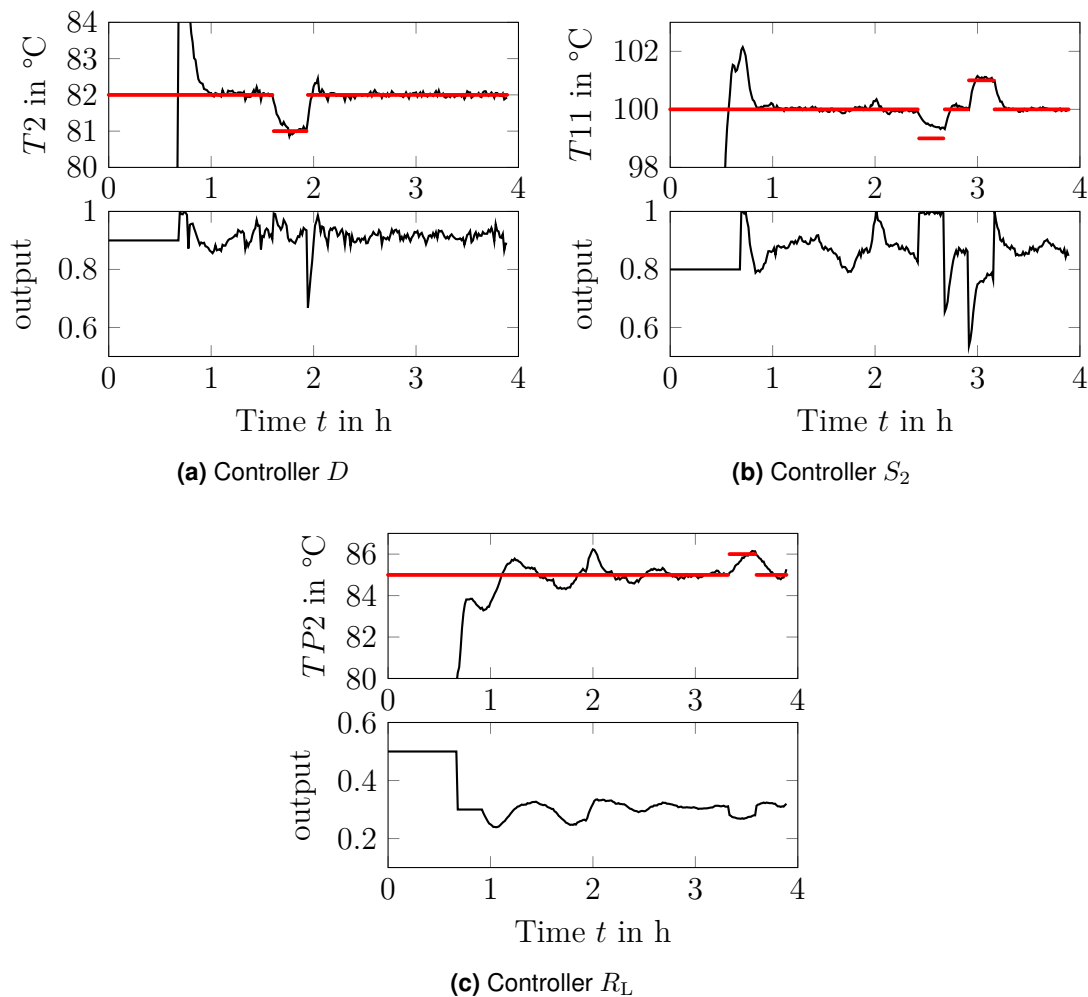


Figure 4.2: Controller outputs and controlled temperatures of D , S_2 and R_L of closed loop experiment with manipulated liquid split and deactivated vapor split

As can be seen in the figure 4.2, several setpoint changes were made during the experiment once the temperature profile was stable. Thus, it was investigated whether the controllers are able to maintain the controlled temperatures. The feed was recirculated after 2.25 h. This caused no observable temperature change.

After starting the column, the feed flow F was started as soon as the heat front reached temperature TP_4 during heat-up. This was the case after 34 min. After 41 min the controller of the upper product stream and the side product stream were started as soon as the heat front reached the top of the column. Simultaneously the liquid split R_L was set to 0.3. Previously this was set to 0.5 to ensure even liquid distribution during heat-up.

The automatic control of R_L was activated after 55 min as soon as the controlled temperature TP_2 was more or less stable. After activating the controllers, it was waited until a stable temperature profile of the column was established and all setpoints were reached.

Several setpoint disturbances were introduced during this experiment. After about 1.5 h the setpoint of controller D was reduced to 81 °C. The controller output increased almost immediately as can be seen in figure 4.2a. The new setpoint of T2 was reached and stable about 10 min after the change was made. The setpoint temperature was changed back to 82 °C after 2 h. This temperature value was reached by T2 immediately. Whereby an overshoot of the controlled temperature TP2 can be observed. After this overshoot the controller D was able to maintain the setpoint. This change of setpoints has an impact on the controlled temperature of the liquid split TP2 and therefore on the output of the liquid split. This can be seen in figure 4.2c. As soon as the setpoint of D is reduced and the temperature T2 starts to decrease, the temperature TP2 starts to decrease as well. To counteract this and ensure the setpoint, the R_L controller reduces the output. Thus, more liquid is fed into the main column and the temperature TP2 starts to rise. The change back to 82 °C of the setpoint of controller D caused a rise of temperature TP2. As a result the controller R_L increased the liquid split to about 0.3. The controller R_L is able to maintain the setpoint temperature.

After about 2.5 h the setpoint of the S_2 controller was reduced to 99 °C. Although the output had reached the maximum, the controller could not reach the setpoint temperature. Therefore the setpoint was changed back to 100 °C 15 min later, which was reached fast with a minor overshoot. Another setpoint change of S_2 to 101 °C was made after about 3 h. This temperature was reached by T11 quickly without an overshoot. Likewise, the change back to 100 °C after 3.2 h was handled by controller S_2 . These setpoint changes of controller S_2 had a similar impact on the liquid split and the respective controlled temperature TP2 as the setpoint changes of controller D . However, the influence is weaker than for the changes of controller D .

Last, the setpoint of controller R_L was changed from 85 °C to 86 °C. To increase the controlled temperature, the output of controller R_L decreased. The controlled temperature T11 reached the setpoint value after about 10 min. Likewise the controller was able to reach the desired temperature after the setpoint was changed back to 85 °C. This took as well about 10 min. Therefore the controller R_L was able to handle the setpoint changes. The setpoint changes of controller R_L had no influence on the controlled temperatures of D and S_2 .

4.2 Vapor split

The active vapor split is introduced in this section. Analogous to the experiments with active liquid split, open-loop as well as closed-loop vapor split experiments are discussed in the following subsections. At first an open-loop experiment with several step changes of the vapor split is discussed. In the second subsection a closed-loop experiment with fully automatic control of the vapor split R_V is discussed. All vapor split experiments were run with S_2 as product sidedraw.

The introduction of the active vapor split brought some problems. As mentioned above, the vapor valves are poorly designed in some respects. The choice of the step range of the vapor valves caused some problems during pre-testing of the valves. At a vapor

split of 0.5, both valves move to the maximum opening step. Is the vapor split lower than 0.5, the valve at the main column is fully opened and the prefractionator valves is closing. If the vapor split is bigger than 0.5, it is the other way around. During pre-testing a vapor split of 0.5 using a step range of 8 already caused a temperature drop in the prefractionator. At this valve opening, the vapor flow should actually not be affected and thus no change in temperature should occur. This indicates that a vapor valve step range of 8 might be too small. In several preliminary tests a vapor valve step range of 10 was identified as an appropriate range.

Another poorly designed component of the valves were the downcomers, which pass the liquid dripping from above through the valves. To ensure a liquid seal to the vapor, the downcomers have a restriction at the lower end. Due to these constrictions, glass fragments of the Raschig Rings from the packing above accumulate in the downcomers. This leads to blockage and thus to an accumulation of liquid on the valves over time. The liquid then flows through the vapor orifices and affects the vapor flow and split. This leads to a shift of the effective vapor valve step range to higher steps. The accumulation of fluid on the vapor valves even caused flooding of the column during certain pre-tests. To solve this problem in the short term, the valves were disassembled and the downcomers cleaned. In the long term, the downcomers must be designed differently.

4.2.1 Experimental run with different step changes of R_V

An open-loop experiment using active vapor split R_V is described and discussed in this section. Similar to the R_L experiments, several step changes of the vapor split were made to investigate how the vapor flow and the temperature in the column are affected. During the discussed run the initially set step range of 8 was used.

Since this experiment was one of the early tests, the upper product cooler was still mounted incorrectly. For this reason, there was a temperature increase during the experiment, especially at the top of the column. The reasons for this have already been discussed in section 4.1.1. Additionally, the temperature T13 was still used as controlled variable for the S_2 sidedraw controller.

The reboiler duty was set to $Q = 1.8 \text{ kW}$ and the feed flow was set to $F = 2.0 \text{ L/h}$. The feed flow was started together with the controllers after the column was heated up. During heat-up the liquid split was set to 0.5. After the column heat-up, the liquid split R_L was set to a fixed value of 0.3 during the whole duration of the experiment. The top product controller used a temperature setpoint of $82 \text{ }^\circ\text{C}$ for the controlled temperature T2 and the controller S_2 used a setpoint of $108 \text{ }^\circ\text{C}$. For the discussed experiment a mixture consisting of $x_{\text{ethanol}} = 0.15$, $x_{\text{propanol}} = 0.55$ and $x_{\text{butanol}} = 0.3$ was used.

The trend of the controlled temperatures of D , S_2 , R_L and R_V together with the respective controller outputs are plotted in the following figure 4.3.

The heat-up of the column was started by activating the reboiler. After the heat-up of the column, the controllers D and S_2 and the feed flow were activated. Simultaneously,

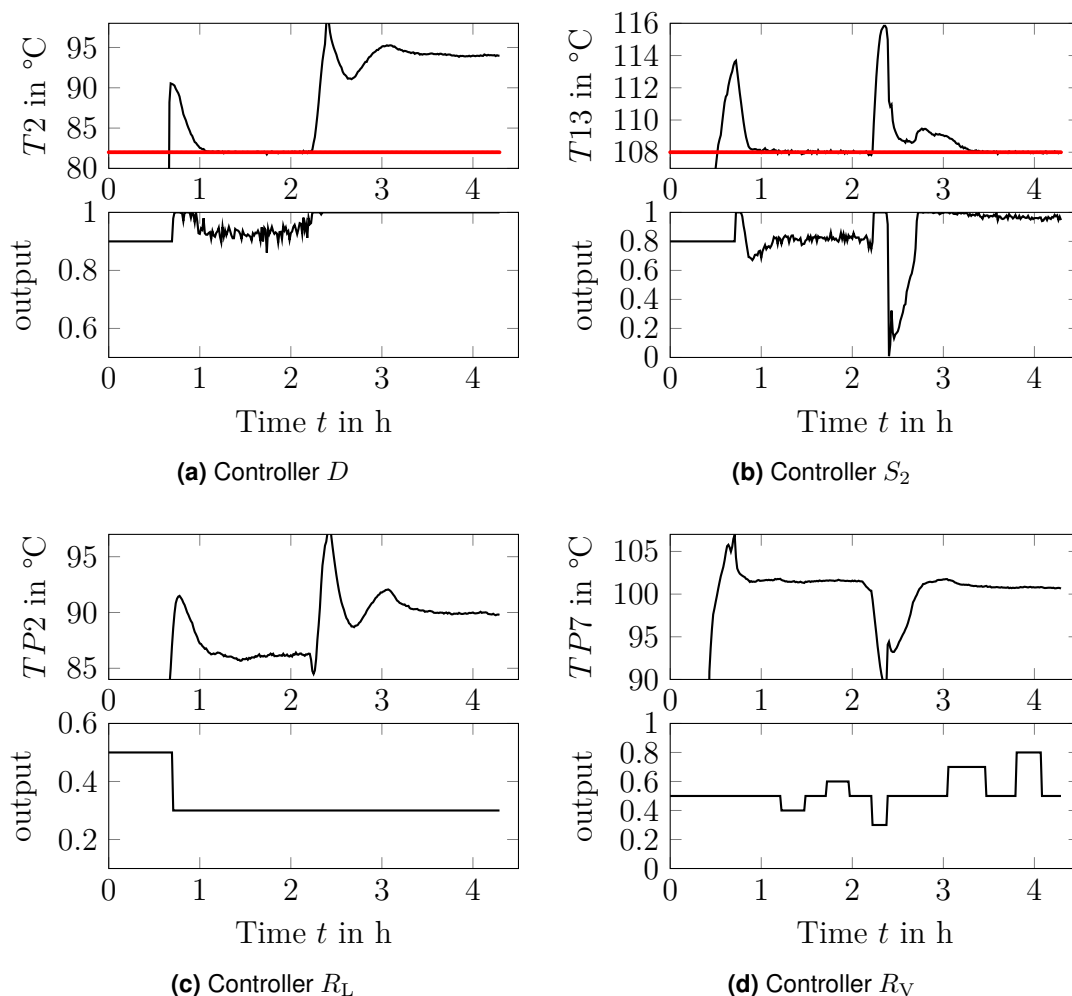


Figure 4.3: Controller outputs and controlled temperatures of D , S_2 , R_L and R_V of open loop experiment with manipulated vapor split and fixed liquid split

the liquid split was set to 0.3. This was the case after 45 min. It was then waited until the temperature profile of the column was stable and the setpoints of the controllers were reached.

After 1.25 h the profile was stable. Therefore the vapor split was activated. At first a vapor split of 0.4 was introduced. This should direct more vapor to the main column and therefore the temperature in the main column should increase and the temperature in the prefractionator (TP7) should decrease. However, no temperature change was observed due to this vapor split. The vapor split was set back to 0.5 after 1.5 h. Therefore both valves were opened to step 8.

The vapor split was set to 0.6 after 1.75 h. This should direct more vapor to the prefractionator and therefore it is expected that the temperature TP7 should rise. As can be seen in figure 4.3d, the temperature TP7 is not affected by the change of the vapor split. The vapor split is set back to 0.5 after 2 h. The feed was recirculated right after this change.

After 2.25 h the vapor split was reduced to 0.3. This caused an immediate response. The temperature TP7 dropped by more than 10°C , which is consistent with the lower

vapor flow to the prefractionator due to the set vapor split. The other controlled temperatures TP2, T2 and T13 show an increase of several degrees in figure 4.3 respectively. This is consistent with the expectations. 10 min after the change to 0.3, the vapor split is set back to 0.5. This causes a slow increase of temperature TP7. It took almost 45 min until the same temperature prior to the vapor split change was reached again. Simultaneously, the other controlled temperatures started to decrease after the vapor split was set back to 0.5. But only T13 was able to reach the temperature setpoint again after about 45 min. TP2 stabilized at a temperature level 5 °C higher than before the vapor split change. The temperature T2 settled at a temperature 12 °C hotter than the setpoint of controller *D*. Even though the controller *D* was at maximum output and no product should be withdrawn, the temperature did not decrease. The reason for this was probably the improperly installed heat exchanger at the top of the column and the resulting continuous flow of product. As a result, a larger quantity of medium boiler accumulated at the top, resulting in an increased temperature.

The feed was recirculated a second time after 3 h. After the temperature profile was stable again, two vapor split changes to 0.7 and 0.8 were performed 3 h and 3.75 h after the start of the experiment. None of these two vapor splits had any observable impact on the temperature profile as can be seen in figure 4.3.

It can be concluded that the vapor split is in principle able to affect the controlled temperature TP7. However, the vapor valve step range must be adjusted. Since most of the vapor split steps had no effect on the temperatures and only a split of 0.3 has shown an effect, it can be concluded that the vapor valves are only effective at the limits of the openings. This illustrates the potential for improvement of the vapor valves. The vapor valve step range is adjusted to 10 in the following experiments. Additionally, it cannot be ruled out that the effect of the improperly positioned cooler influenced the performance of the vapor split. The product heat exchanger at the top is fixed for the following runs.

4.2.2 Experimental run with fixed liquid and controlled vapor split

The closed-loop experiment using fully automatic control of the vapor split R_V is discussed in this subsection. Similar to the closed-loop run with control of the liquid split, several setpoint changes of the R_V controller were performed. The response of the temperature profile to this setpoint changes was investigated.

Again, the reboiler duty was set to $Q = 1.8$ kW and the feed flow to $F = 2.0$ L/h. The feed input was started as soon as the temperature at the inlet TP4 reached about 80 °C during the heat-up. As with all measurements, a liquid split of 0.5 was set during heating. After the column was heated, a fixed liquid split R_L of 0.3 was set for the duration of the experiment. The controller *D* at the top of the column used a setpoint of 82 °C for controlling temperature T2. The sideproduct draw S_2 used T11 as controlled temperature with a setpoint of 100 °C. The vapor valve step range was set to 10. A mixture consisting of $x_{\text{ethanol}} = 0.25$, $x_{\text{propanol}} = 0.45$ and $x_{\text{butanol}} = 0.3$ was used in this experiment.

The plots of the controller outputs and the controlled temperatures are respectively plotted over time in figure 4.4.

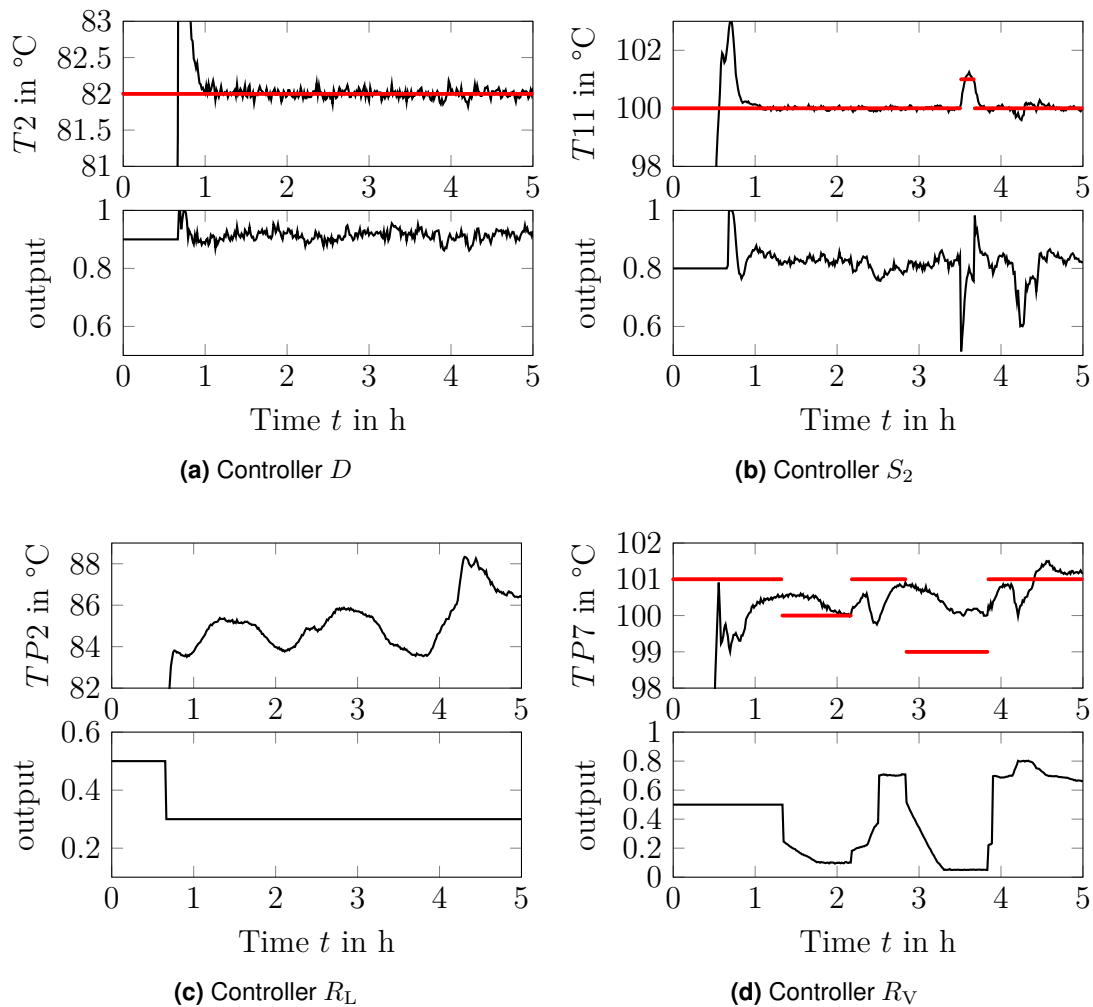


Figure 4.4: Controller outputs and controlled temperatures of D , S_2 , R_L and R_V of closed loop experiment with manipulated vapor split and fixed liquid split

The feed flow was started 33 min after the activation of the reboiler. After 39 min the controllers of the top and the sideproduct were activated and the liquid split R_L was set to a fixed value of 0.3. After that, it was waited until the temperature profile was stable. As in figures 4.4a and 4.4b can be seen the setpoint temperatures of the controllers D and S_2 could be reached and maintained almost perfectly over the whole duration of the experiment.

As soon as the temperatures were stable, the vapor split R_V was activated after 1 h 10 min. The vapor split was set to 0.5, thus both valves were closed to step 10. It was waited for 10 min if this already had any impact on the temperatures. This was not the case. After 1.3 h the automatic control of R_V was activated. A setpoint of 100 °C for controlled temperature TP7 was used. A controller output of 0.25 was used as starting point of the controller.

The setpoint temperature was reached about 40 min after the control was activated by the vapor split controller. This can be seen in figure 4.4d. The vapor split controller

was thus able to control the temperature TP7. However, the figure 4.4d shows that the controller had little influence at first. Only when an output of about 0.1 was reached, an influence on the temperature TP7 became apparent. This again shows that the vapor split controller is only able to effectively influence the vapor flow near the limits of the controller output. The decrease of the temperature TP7 caused a decrease of temperature TP2 as well. Since this temperature was not controlled, the fluctuations could be clearly observed. This indicates that the controllers R_L and R_V may influence each other if they are both activated.

After 2 h 10 min the setpoint of controller R_V was changed to 101 °C. This resulted in an immediate increase of the controlled temperature TP7 as well as of temperature TP2. The feed was recirculated after 2.3 h, this led to a drop of temperature TP7. This can be explained by a slightly different composition of the feed due to the recirculation. The amount of heavy boiler that had to be returned could only be estimated using a sight glass at the reboiler. Accordingly, it was difficult to ensure the exact same composition each time. Nevertheless, the controller was able to compensate for this unintentionally introduced disturbance. The R_V controller output was reset to 0.7 after 2.5 h. The setpoint of 101 °C was reached after about 2.8 h.

Another setpoint change to 99 °C was made after 2 h 50 min. This setpoint could not be reached even when the controller output was at the lower limit of 0.05. A setpoint change of the S_2 to 101 °C after 3.5 h was made to try to reach the setpoint of temperature TP7. This did not show the desired effect. Therefore the S_2 setpoint was changed back to 100 °C after 10 min.

After 3 h 50 min the setpoint of the vapor split controller was set to 101 °C. Simultaneously, the output of the controller was reset to 0.7. This caused an immediate increase of the controlled temperature TP7 and temperature TP2. After about 4 h the feed was recirculated again. This again caused a drop of TP7. The controller R_V was able to handle this disturbance as well. The setpoint was reached after a slight overshoot after 4.8 h. The experiment was completed after 5 h.

This experiment showed that the control of the temperature TP7 with the help of the vapor split is possible despite the poorly designed vapor valves. Although the vapor split controller responds much more slowly than the D and S_2 controllers, the setpoints were still reached sufficiently quickly and accurately. Once again, it was observed that the vapor valves are only able to effectively influence the vapor flow near the opening limits. In addition, an occasional reset of the controller output was necessary. This could probably be fixed by tuning the controller. Another issue which should be fixed is the variation of feed composition due to the recirculation. This could be resolved by more accurately measuring the volumes of the three product streams.

4.3 Vapor and Liquid split control

The next step is to introduce a 4-point control structure with simultaneous temperature control of liquid split, vapor split, top product flow and side product flow. The goal of this control structure was to improve the control of the column by using two different

temperatures in the prefractionator as controlled variables in two separate loops. The speed of stabilization of the temperature profile should be increased with this control arrangement. Several experiments using this control structure were conducted. The description of all experiments would go beyond the scope of this paper. For this reason, only one representative run will be described and discussed in this section.

A reboiler duty of $Q = 1.8\text{ kW}$ and a feed flow of $F = 2.0\text{ L/h}$ were used in the discussed experiment. Again, the feed was activated as soon as temperature TP4 reached about $80\text{ }^\circ\text{C}$ during heat-up. A feed mixture of $x_{\text{ethanol}} = 0.25$, $x_{\text{propanol}} = 0.45$ and $x_{\text{butanol}} = 0.3$ was used in the run. The step range of the vapor split valves was set to 10 for this experiment as well. As soon as the top of the column was heated up, the controllers were activated one after another. The controller D used a setpoint for temperature T2 of $82\text{ }^\circ\text{C}$ and the controller S_2 used a setpoint of $100\text{ }^\circ\text{C}$ to control temperature T11. For the control of temperature TP2 using the liquid split as manipulated variable a setpoint of $85\text{ }^\circ\text{C}$ was used. The controller R_V used a setpoint of $100\text{ }^\circ\text{C}$ for the control of temperature TP7. Several setpoint changes of controller R_V were made during this experiment. The plots of the controller outputs and the controlled temperatures are plotted over time in figure 4.5.

The feed flow was activated 32 min after the start of the heat input. After 38 min the automatic control of D and S_2 was activated. Simultaneously, the liquid split R_L was set from 0.5 during heat-up to 0.3. The vapor split valves were still fully opened at step 50 at this point. After 50 min the fully automatic control of the liquid split was activated. Afterwards, it was waited until the temperature profile was stabilized.

The vapor split R_V was activated after 1.25 h. At first a fixed value of 0.5 was set. Therefore both valves were closed to step 10. It was waited to make sure that this would not already have an impact on the temperatures, as has been observed in previous runs. No temperature changes were observed after activating the vapor valves. Therefore the fully automatic control of the vapor split was activated after 1 h 25 min. At first a setpoint for the controlled temperature TP7 of $100\text{ }^\circ\text{C}$ was implemented. This setpoint was reached after about 2.2 h.

After about 2.25 h the feed was recirculated from the product tanks. This caused a drop of temperature TP7 shortly after the recirculation. This is shown in figure 4.5d. Again, this can be explained by a slightly different feed composition due to the recirculation. The controller was able to handle this disturbance and to maintain the setpoint of the controlled temperature. The setpoint temperature was reached again after 2.5 h. At the same time, the temperature TP2 also showed a decrease. This could be handled by the respective controller R_L using the liquid split. Which led to a decrease of the liquid split and therefore to more liquid in the main column.

A setpoint change of the controller R_V was made after 2 h 35 min. The setpoint was changed to $99\text{ }^\circ\text{C}$. Consequently, the controller R_V reacted and the valve of the prefractionator was closed further, so that more vapor was fed into the main column. This caused a decrease of temperature in the prefractionator, which can be seen in figures 4.5c and 4.5d. Thus, the newly set setpoint was reached.

After 2 h 55 min the setpoint for the R_V controlled temperature TP7 was changed

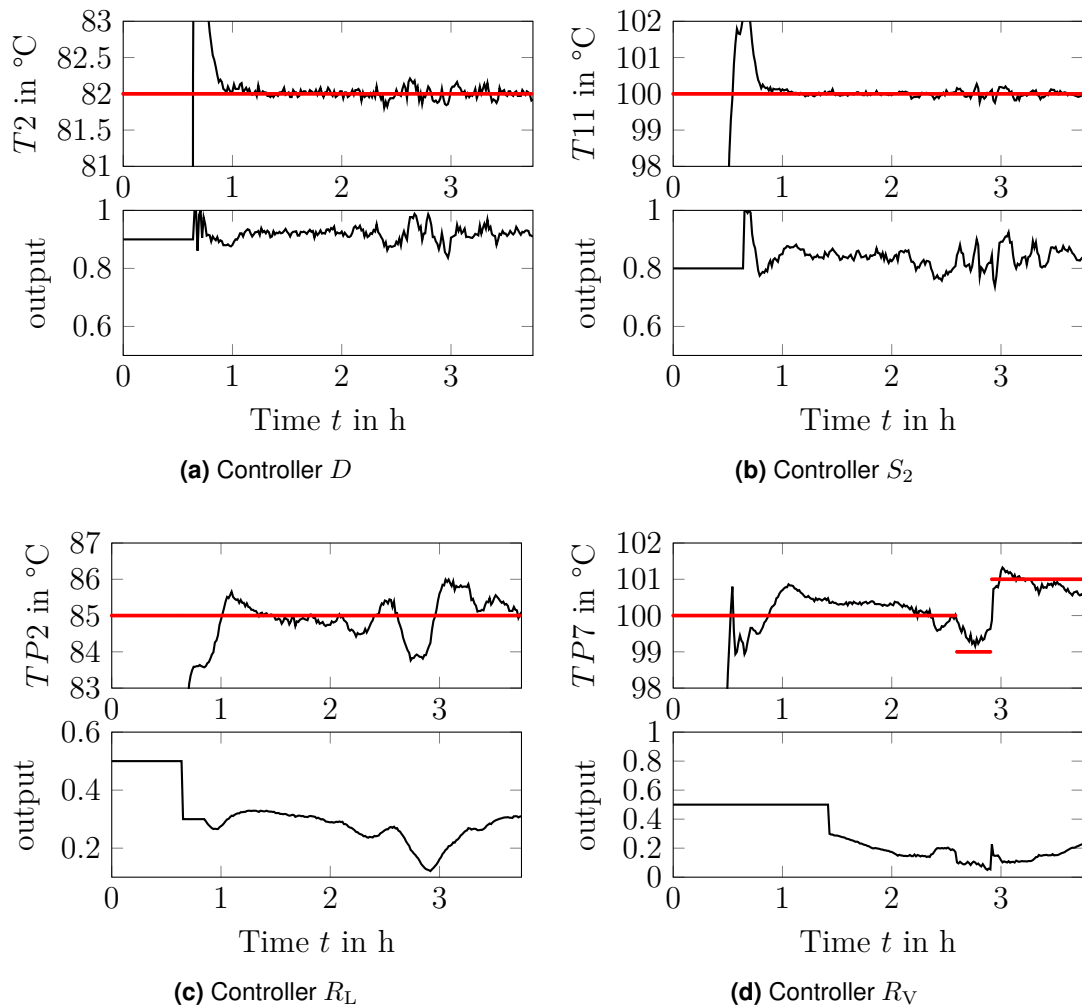


Figure 4.5: Controller outputs and controlled temperatures of D , S_2 , R_L and R_V of closed loop experiment with 4-point control structure

again to $101\text{ }^\circ\text{C}$. This setpoint was reached almost immediately. Whereby an overshoot of temperature TP7 was observed. This temperature setpoint could be more or less maintained for the rest of the experiment. This setpoint disturbance had an impact on the liquid split controlled temperature TP2. As soon as the temperature TP7 increased, the temperature TP2 increased as well. This could be handled by the R_L controller by redirecting more liquid to the prefractionator. This corresponds to an increased liquid split.

The controllers D and S_2 were hardly affected by the setpoint changes of controller R_V and could always maintain their setpoints. Only minor deviations can be seen in figures 4.5a and 4.5b.

The experiment showed that the controllers of R_V and R_L can influence each other. The introduced setpoint disturbances could be compensated for by the controllers with sufficient speed and accuracy. The setpoint temperatures of all controllers could be maintained. Nevertheless, further experiments are necessary to validate the performance of this control scheme.

5 Summary and Outlook

The results of the internship and the thesis are summarized in this chapter. In addition, an outlook for future work and possible improvements of the column setup are given. The main goals of the research internship were achieved. Thus, the control software in *LabView* was updated to a newer version, as was the operating system. In each case, it was not possible to update to the latest version because the *FieldPoint* hardware is not compatible with the latest versions. Additionally, some improvements such as a safety switch for to high reboiler temperatures were implemented in the control software.

The main goal was to bring the Kaibel column back into operation, which was also accomplished. After some small adjustments and maintenance works, the column was operated with a three-component mixture of ethanol, propanol and butanol. Several test runs were carried out to check the operation of the pilot plant and to familiarize with the control of the column.

Unfortunately, some issues caused significant time delays during the column testing phase. For example, a malfunction in a control box and the subsequent wait for delivery of spare parts led to an almost three-week shutdown of the column. As a consequence, not as many experiments could be performed on the Kaibel column as hoped. Nevertheless, some experiments could be performed and meaningful results could be obtained. In addition, the most important goal of recommission of the pilot plant was achieved.

Several experiments with different temperature control schemes were conducted. The flows of the top and side product were controlled during every performed run. Control schemes with a three-point control using the liquid split or the vapor split as manipulated variables and temperatures in the prefractionator as controlled variables were investigated. These control arrangements were able to maintain the temperature setpoints and to achieve a stable temperature profile. Whereas it was observed that the control of vapor and liquid split is slower than the control of product streams. In addition, it was found that the vapor split can only effectively influence the vapor flow and thus the controlled temperature at the limits of the controller output. Additionally a four-point control scheme with simultaneous control of liquid and vapor split was investigated. This control scheme was able to stabilize the temperature profile of the Kaibel column as well. Several setpoint changes of the controllers were introduced during the experiments. Most of these changes could be handled by the investigated control structures. Nevertheless more experiments would be necessary to reproduce and validate the results which were obtained so far.

There are several issues that need to be fixed and several improvements that should be added to the Kaibel column pilot plant in future works. First of all, the design of the vapor split valves should be reworked. Based on corresponding observations during

various test runs, it is assumed that glass fragments of the Raschig Rings accumulate inside the downcomers of the valves. Due to the narrowed opening of the downcomers, this leads to a buildup of liquid on the valves and thus to impaired valve performance. Therefore the downcomers should be redesigned so that any pieces of glass can pass through and still ensure a sufficient liquid seal. A proposal for a possible downcomer design is illustrated in figure 5.1 where blue lines represent the fluid flowing from above.

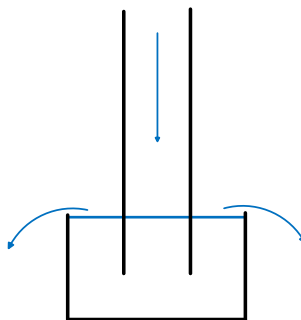


Figure 5.1: Proposal of alternative design for a downcomer for the vapor split valve

As suggested by the illustration 5.1 a downcomer without a narrowing at the end could be installed. Therefore any glass fragments can pass the downcomer without problems. To ensure a sufficient liquid level as seal to the vapor, a cup could be installed at the lower end of the downcomer which would hold a certain liquid level.

Additionally, the opening steps of the vapor split valves should be redesigned. The step size of the valves should be refined in the effective range, so that more precise settings are possible. This could be done by refining the rack and pinion as well as the stepper motor. With more precise step size the control of the vapor split may be more accurate. Furthermore, analytic measurements to testify the purity of the products should be conducted in future works. This could be used to check if the column is able to achieve pure product fractions and thus validate the performance of the temperature control. Measurements of samples of the product streams using gaschromatography would be suitable for this purpose.

As already mentioned the controller of the vapor split is only effective near to the limits of the controller output. Therefore it would make sense to re-tune the vapor split controller to achieve quicker responses and a wider effective range of the controller output. Whereby this problem could already be solved by a redesign of the valve step sizes as already mentioned. It must be mentioned that the used controller parameters have been taken directly from KORBELAROVA 2017. It is therefore possible that they are not suitable for the used three-component mixture. Thus, a systematic tuning of all controllers would make sense. Especially for the liquid and vapor split controllers a systematic tuning would probably cause quicker responses.

A Appendix

A.1 Plots of reproduction measurements

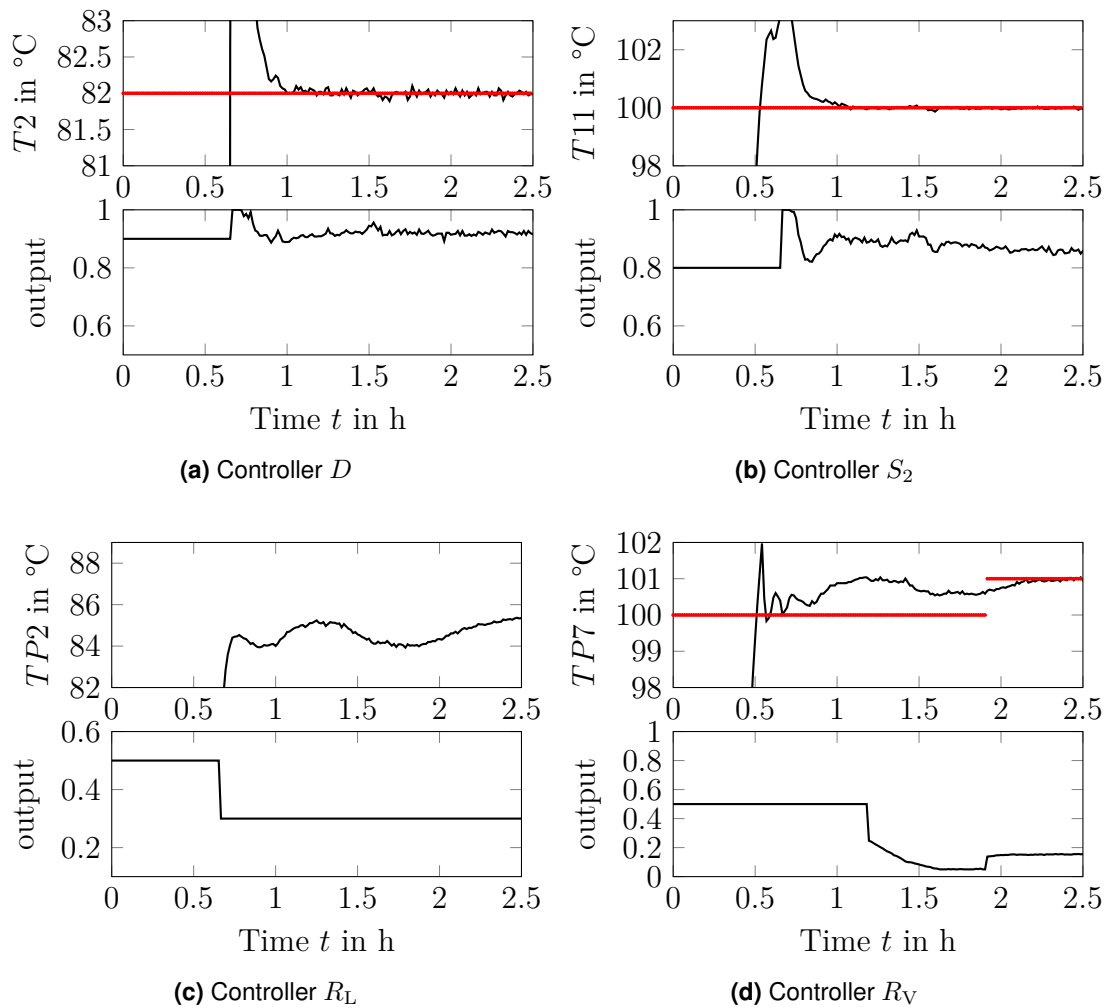


Figure A.1: Reproduction measurement of experiment discussed in section 4.2.2, Controller outputs and controlled temperatures of D , S_2 , R_L and R_V of closed loop experiment with manipulated vapor split and fixed liquid split

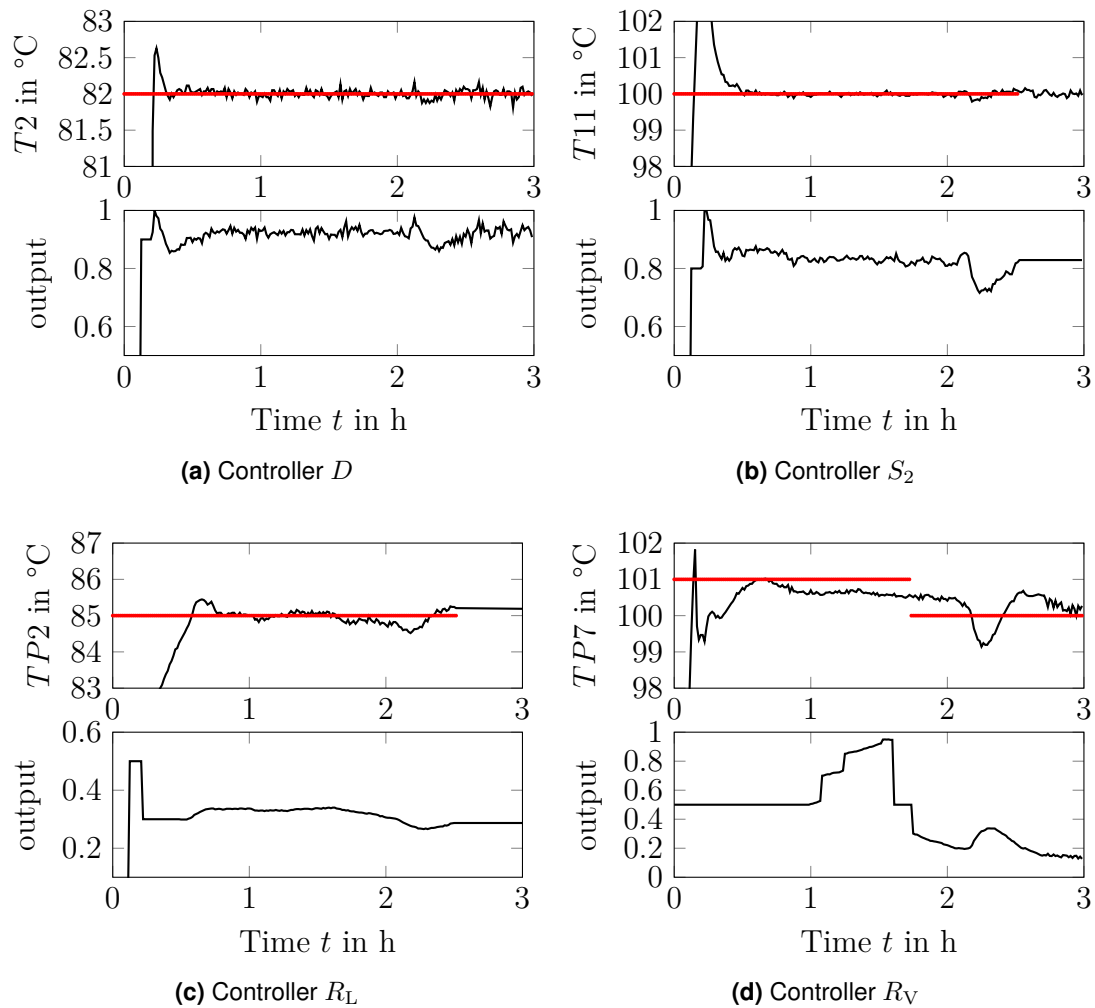


Figure A.2: Reproduction measurement of experiment discussed in section 4.3, Controller outputs and controlled temperatures of D , S_2 , R_L and R_V of closed loop experiment with 4-point control structure

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