

Laboratory Runs on Kaibel Column - Process Review

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Problem Description

The goal of the project was to carry out experiments on pilot Kaibel column to test it's operation after shutdown, and recent rebuild.

The project work consists of following parts:

- Set up standard procedure for working with the column
- Run experiments to verify controller settings, and overall performance
- Evaluate risk assessment for Kaibel column
- Summarize challenges connected to practical operation of the column.

Preface

This is the report for the Chemical Engineering, Specialization Project (TKP4580). The work was carried out during autumn semester 2017 on Department of Chemical Engineering at Norwegian University of Science and Technology (NTNU). The project is part of my master's degree in Chemical Engineering at NTNU. This report could work as an introduction to operating of experimental Kaibel distillation column, covering the description of the instrument, setting standard operational procedures, evaluating risks, and summarizing some of the challenges met during operation.

Trondheim, 2017-12-19

Johana Korbearova

Summary

The project focus on experimental work performed on Kaibel column in laboratory Hall C at the Chemical Engineering Department, NTNU. The main motivation behind using Kaibel column are potential energy savings compare to traditional distillation columns.

The experimental Kaibel Column is described, together with it's control structure. Kaibel column is controlled by 4 temperature control loops.

The standard procedure for running the experimental Kaibel column is set. The risk assessment is performed covering all significant hazards. The practical challenges met during operation are summarized, together with proposed solution. The results of experiments verifying the column operation, and controller settings are presented.

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Introduction

Distillation is a preferred solution for separation of liquid mixtures. Distillation process is highly energy consuming, and it is desirable to minimize necessary energy input.

One of the solutions to reduce energy consumption was introduced by [Kaibel \(1987\)](#). Kaibel column is a thermally coupled dividing wall column, which enables separation of 4 components in one column. As it is shown on figure 1.1 (from [Dwivedi \(2013\)](#)), feed mixture of 4 compounds enters the column on the left entering prefractionator section, where separation of two lighter and two heavier components takes place, the separation is then finished in the main part of the column, where the products are obtained. Compared to conventional sequence of 3 binary distillation columns for separation of 4 components, see figure 1.2 (from [Kvernland \(2009\)](#)), the Kaibel column requires only one reboiler and one cooler. If the Kaibel column is compared to conventional sequence of binary distillation columns this arrangement reduce energy about 30 %, referring to theoretical study of [Halvorsen and Skogestad \(2006\)](#).

However, the energy savings are strictly connected to operation mode of the column. If the column does not run optimally, these savings could be completely neglected.

The separation in one-shell-column such as Kaibel also provides significant the capital savings compare to sequence of distillation columns.

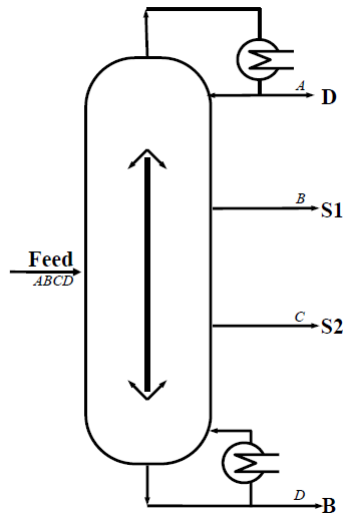


Figure 1.1: Diwiding wall implementation of Kaibel column. Enables separation of 4 compounds. Set up requires one reboiler and one cooler (Dwivedi, 2013)

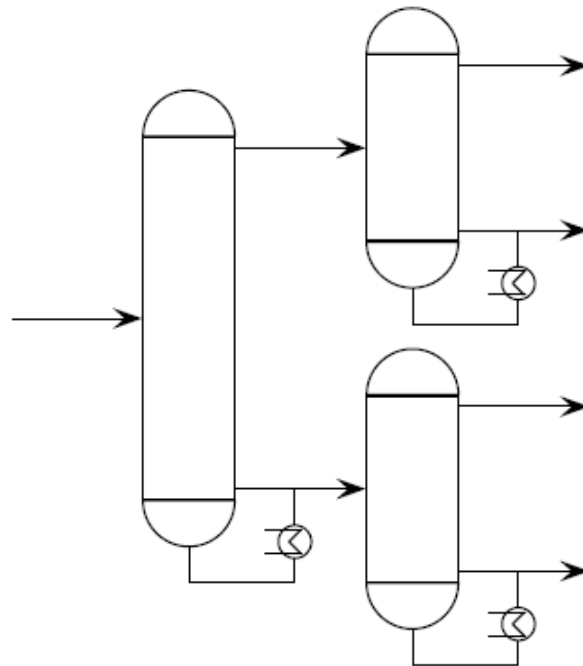


Figure 1.2: Conventional sequence of binary distillation columns for separation of 4 compounds. Set up has three reboilers and three coolers. Kvernland (2009)

Experimental Setup

2.1 Kaibel Column

The experimental column situated in laboratory Hall C at the Chemical Engineering Department, NTNU is shown in figure 2.1, [Strandberg \(2011\)](#). Kaibel column is realized as a two-shell, although this geometry is thermodynamically equivalent to dividing wall column. This column was built by ([Strandberg, 2011](#)) during his Phd. studies at NTNU, more detail information about column could be found there.

The column is made of glass sections produced by Normag Labortechnik in Germany. The individual sections are connected by flanges. Flanges are attached to aluminum frame by springs, which lowers the pressure on sections bellow, and secures column position.

As seen in the figure 2.1 ([Dwivedi, 2013](#)), the column consist of 7 sections, sections 1 and 2 (left branch) represents prefractionator, while 3 to 7 the main column. The sections are vacuum coated, and covered by silver to minimize heat loss. The inner diameter of sections is written in table 2.1.

Column is packed by glass Raschig rings with diameter 6 mm. The packing high varies in column sections, values are summarized in table 2.1. The height equivalent of a theoretical plate (HETP) was estimated by [Dwivedi \(2013\)](#) to be 16 cm for this column.

The valves ensuring the liquid split and product withdrawal are swinging funnels operating

Table 2.1: Specification of column sections [Kvernland \(2009\)](#) [Dwivedi \(2013\)](#)

Section	Inner diameter [mm]	Packing height [m]
-		
1	50	1.10
2	50	1.60
3	70	0.65
4	50	0.65
5	50	0.65
6	50	0.75
7	50	0.90

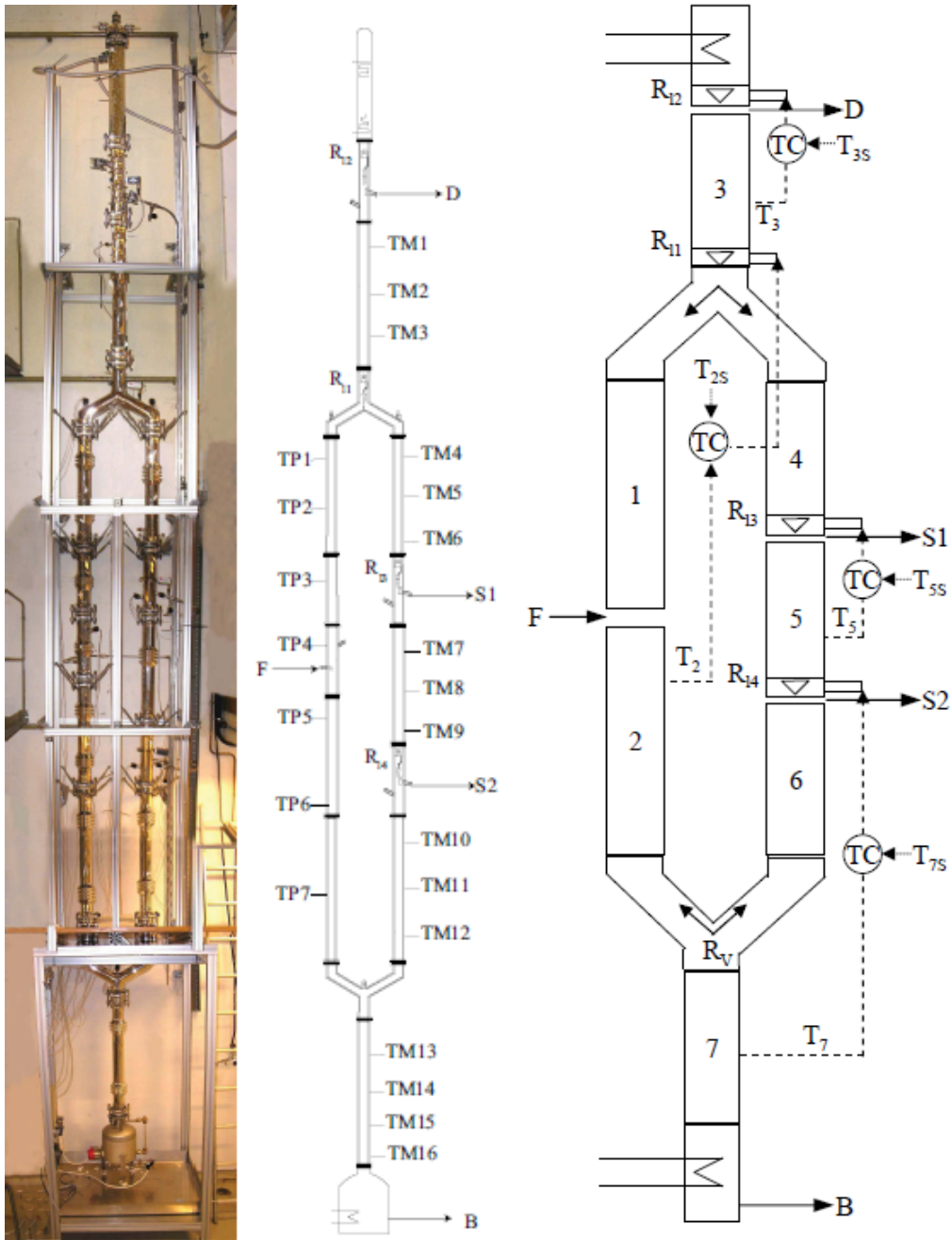


Figure 2.1: Picture of experimental column
 Picture of experimental column
 Schematic of location of temperature sensors
 Schematic of proposed 4-point temperature regulatory control structure

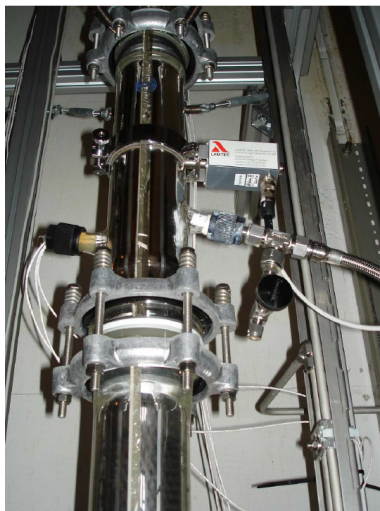


Figure 2.2: Picture of side stream product withdrawal. Swinging funnel leads the liquid from collector outside the column as a product, or back as a reflux.

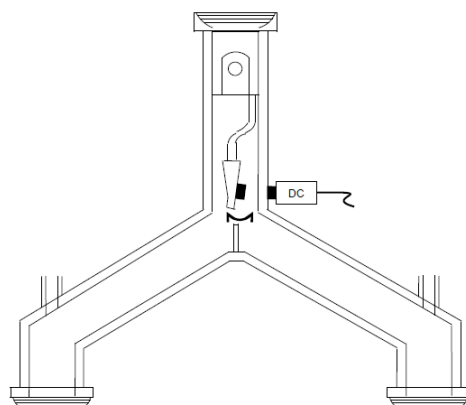


Figure 2.3: Schematic of liquid split. Swinging funnel leads liquid flow according to solenoid to pre-fractionator (left), or to main column (right).

at ON/OFF states controlled by externally placed solenoids. The positions of funnel are changing with given period to ensure preferred liquid split, and stable liquid flow in the column.

The side stream withdrawal is shown on figure 2.2, the swinging funnel inside directs the liquid flow outside the column as a product, or back as a reflux.

The liquid split section is shown on schematic, figure 2.3, liquid is accumulated in collector, the funnel directs the liquid flow according to solenoid either to pre-fractionator on the left, or to main column on the right.

This column enables usage of vapor split as a manipulated variable. This is facilitated by two vapor split valves developed by Strandberg (), see figure 2.4. Valves are operated by external electrical motors.

The schematic of vapor split valve is presented in figure 2.5. The rack and pinion assembly

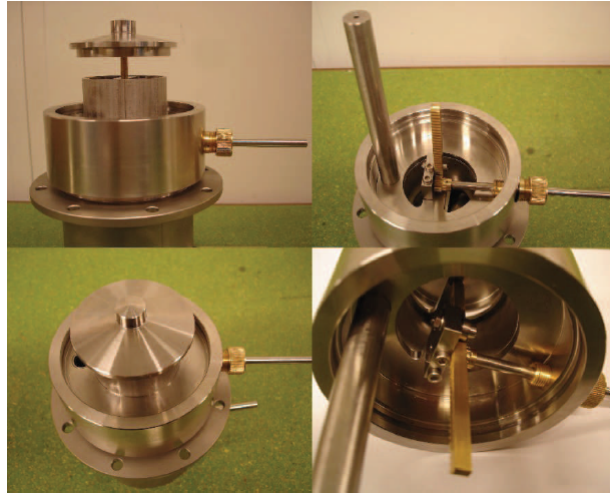


Figure 2.4: Picture of vapor split valve.

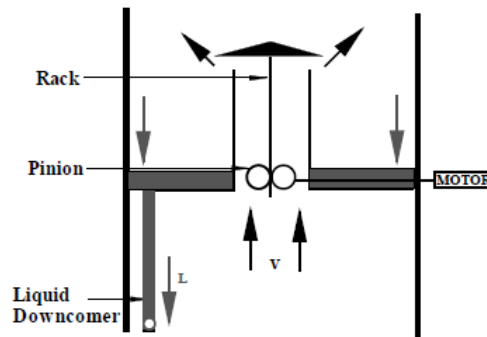


Figure 2.5: Schematic of vapor split valve. The vapor flow is changed by adjusting cap position through rack and pinion by electric motor.

is used, the vapor flow is adjusted by changing the cap position, the liquid downcomer allows liquid to flow against vapor. Two valves are situated in lower parts of section 2 and 6 in pre-fractionator, and main column, respectively.

The side stream coolers are installed to lower the temperature of leaving product. The liquid seal on product stream, following coolers, avoids the leakage of vapor through side stream, which would cause disturbance to the process. This also works as indicators of pressure drop in respective column section. See figure 4.1.

The reboiler is kettle type electric boiler with maximal energy input 2.9 kWh. Energy input is adjusted by thyristor. Reboiler capacity is 15 l, while operation minimum is 3 l. Since the level control on reboiler is not implemented, the bottom product is kept accumulating in reboiler during experiments.

The top of the column is equipped by condenser cooled by water. The condensed product liquid flows back to column to split valve where it is divided to distillate and liquid reflux.



Figure 2.6: Picture of liquid seal positioned on product stream. It ensures that the vapor is not leaking through product stream.

The feed is pumped into the system by a digital diaphragm dosing pump. Possible flow range is 0.2-20 l/h. The electric heater is installed alongside the feed tube to preheat feed, and lower the disturbance caused by cold liquid entering the column.

The column is operated at atmospheric pressure, the total pressure drop under normal operation is about 0.016 bar.

The separation of equimolar mixture of primarily alcohols is studied. The feed mixture consist of methanol, ethanol 1-propanol and 1-butanol.

2.2 Control Structure

According previous studies published by Skogestad and Strandberg [Strandberg \(2011\)](#), it is possible to use 4-point-temperature control for controlling 4-compound distillation columns avoiding 'drift' of product concentration.

The control was performed using decentralized control as shown by Dwivedi (), the other possibility is to use model predictive control (MPC), model for Kaibel column was derived by

Kvernland ().

The decentralized control structure would be further described there, as it was implemented in used LabVIEW.

The control consist of 4 feedback control loops. Temperature controllers enables fast and accurate control of the composition. The most sensitive temperatures in individual sections were chosen as controlled variables. The manipulated variables (MVs) are split valves (resp. swinging funnel ratios) on top product, and side stream products, liquid split valve, and vapor split valve.

Only four of these MVs are necessary to control loops, this extra degree of freedom could be used for optimization purposes, together with column boilup, which is also unused for control.

Control structure is shown on second schematic of figure 2.1.

The 4 control loops are:

1. TP5(T2) controlled by liquid split valve, RL, (or possibly by vapor split valve, RV)
2. TM3(T3) controlled by destilate split valve, RD
3. TM8 (T5) controlled by side stream 1 split valve, RS1
4. TM14 (T7) controlled by sude stream 2 split valve, RS2

The actual manipulated variables are liquid split ratios defined bellow in equations. L refers to flow in individual secitons defined in figure 2.1.

$$RL = \frac{L_1}{L_3} \quad (2.1)$$

$$RD = \frac{L_3}{L_3 + D} \quad (2.2)$$

$$RS1 = \frac{L_5}{L_5 + S1} \quad (2.3)$$

$$RS2 = \frac{L_6}{L_6 + S2} \quad (2.4)$$

Extra feedback control loop is adjusting the temperature of preheater on the feed according to setpoint.

Risk Assessment

For every experimental work is necessary to evaluate risk factors, and possible accidents prior the laboratory work. It helps to remove or control risks, and it is obligatory to know how to proceed in case of accident. The sources of hazard in this case are used chemicals, high operating temperatures, and work in heights. Main risks connected with operation of Kaibel column are described bellow.

3.1 Handling Chemicals

Contact with chemicals is relatively limited during normal operation. Although, the feed mixture circulates in the system, it could be necessary to add some more into feed tank. Sometimes, it could be chosen to take samples of products. The most common contact with chemicals is handling with feed pump during start-up.

Because spill of the chemicals can occur, the protective equipment (gloves, goggles and coat) has to be wear.

According to safety data sheets, used alcohols could be irritating when exposed to skin, get inside eyes, or when breathed in. Then, it is necessary to clean the skin by water, rinse eyes by water, and go to fresh air respectively. The spills on floor or equipment has to be cleaned.

3.2 Leakage of Fluids

Small leakage of the liquids, or vapor could occur during operation. Bigger amounts signals problems, for example breakage of the column.

In this case, it is necessary to shutdown the column, either by turning off heat sources (re-boiler, feed heater, feed flow), or using emergency button on 1st or 2nd floor. Later, find the source of leakage, and make maintenance actions.

It is necessary to observe the column regularly during operation for possible leakages.

The health risk involves exposing to chemicals (as described above), and burn injuries due to contact with hot fluid or equipment.

3.3 Breakage of Equipment

Since the column is made of glass, it could be broken if not carried carefully, or by accident.

Special attention has to be paid during maintenance actions. It is described in maintenance procedure, main points are that the column parts below working place should be covered by padding, and the tools and equipment should be carried in special box.

The breakage could occur by accident, due to falling objects from upper floors. If this happens during operation, it is necessary to provide the immediate shutdown.

The glass fragments could cause injuries, it is necessary to provide first aid.

3.4 Risk of Fire

The risk of fire is always present, since alcohols are flammable compounds. This risk could have external and internal source.

External fire somewhere in the building could not be influenced. Fire could be caused by column, electrical devices could sparkle and alcohols could catch the fire, this accident can not be prevented, the regular checks of the equipment could help.

The possible fire sources are electric heaters, namely reboiler and feed heater. It is necessary to ensure that the amount of liquid is always sufficient. In case of reboiler, the liquid level has to be high enough to ensure safety operation. In case of feed heater, the flow of the feed has to be high enough. Both level, and feed flow into the column should be checked regularly during operation.

In all cases, the action would be to shutdown the column using emergency button, and leave the laboratory.

Health risk involves burn injuries, and exposure to chemicals.

3.5 Risk of Explosion

The explosive properties of alcohols, are not significant compared to the size of the hall where the column is situated. Even with immediate evaporation of all liquids, it would not reach the lower explosion limit.

Calculations prove that the explosion can not occur. With the volume of the air in Hall C equal to 9000 m³, and the total volume of all liquids in the system equal to 15 l, if everything evaporates, the volumetric fraction would be $1.6 \cdot 10^{-5}$. Compared to the most explosive compound, 1-butanol, and its lower explosion limit equal to 0.014, then the actual concentration is more than 800 times lower. It can be concluded that the explosion can not occur.

Risk analysis was evaluated in Health Safety and Environment system at NTNU. The report could be found at <https://avvik.ntnu.no>, under the name “PSE, Instrument Kaibel distillation column, Hall C“, ID 23784. The safety data sheets of chemical compounds, together with Instrument Card are attached to this report.

Operation of Kaibel Column

In this chapter, the practical operation of the Kaibel column is described. The standard procedure is set as a step-by-step manual covering important actions during different operation modes, namely start-up, shut-down, and maintenance actions.

4.1 Start-up Procedure

Following steps describe desired procedure:

- Observe the column for leakages (mainly product storage tanks, feed tank, reboiler). Check if the levels of liquids in reboiler and feed tank are sufficient to enable safety operation, if not turn on the feed to obtain desired the level, resp. empty the product tanks, eventually add new feed mixture to storage tank.
- Turn on feed pump, set it to manual mode, switch to outer hose to check the pump performance, and remove air trapped inside the pump. Switch back to inner circuit, and observe the column feed to ensure operation.
- Turn on electrical enclosure both outside and inside (blue relay) and PC. The column control system is operated by user interface in LabVIEW. Interface is shown in figure 5.1.
- Open the valves of cooling water for cooler (first outlet, then slowly inlet), and open the valve for cooling of side stream products, ensure to obtain stable flow without bubbles.
- In LabVIEW interface at PID settings window set the values of constants for PI(D) controllers as well as set the minimal and maximal values of manipulated variables, feed and reboiler duty, and preheater duty. At PI-controllers window controls the manipulated variables, the steady state temperatures for controlled variables are set there. See proposed values of in table 4.1.
- Make new data file and start saving.

Table 4.1: Settings for Kaibel column: Limit values and PI Controller settings

Name	MV	Min - Max	Kp	I	CV	Setpoint [°C]
D	RD	0 - 1	-0.3	4	TM3(T3)	70
RL	RL	0.3 - 0.7	-0.03	4	TP5(T2)	85
S1	RS1	0 - 1	-0.18	4	TM8(T5)	90
S2	RS2	0 - 1	-0.32	4.16	TM14(T7)	112
Feed flow	-	0- 10	0.65 -	-	-	-
Reboiler	-	0 - 2	-	-	-	-
Feed Heater	inversed normalized input	1 - 0	-0.03	10	Feedheat1	30-60

- To heat up the column, all controllers are in manual mode. set the reboiler duty to 2kWh (maximal value is 2.9 kWh), the feed flow is low. The product split valves are in total reflux position (equal to 1), or little lower. The liquid split valve is 0.5 to ensure even distribution.
- The whole column would eventually heat up, the control is to be switched to automatic mode gradually, starting from top with distillate, once the controlled temperatures are closed to desired steady state values. The feed flow is to be added as well.
- Once the feed flow is relatively high, the preheater could be added to lower temperature disturbance caused by feed, gradually increase the steady state temperature of heating device (from 30 to 60°C), while operating the controller in automatic mode for whole time.
- Once the column is stabilized at steady temperature profile, the experiments are to be cared.

4.2 Operation – Shut-down Procedure

Following steps describe shut-down procedure:

- Stop the heat inputs into the system – feed heater, feed, reboiler. First, turn off feed heater, turn off feed flow, and turn off reboiler. All other controllers can be turned off.
- It is safe to turn close cooling water input approximately after 15 minutes.
- Stop saving in data file, close LabVIEW interface without saving the changes. Download data, and turn off PC, and electrical enclosure.
- Turn off feed pump, it has individual energy source.
- Use emergency button to ensure the electricity is off the system.



Figure 4.1: User interface in LabVIEW used for operation of Kaibel column.

4.3 Operation – Emergency Shut-down

If there is problem indicated, such as leakage of vapor or liquid, high pressure change, or drop in temperature profile, the column has to be stopped immediately.

For operator it is possible to follow shut-down procedure, remove heat source etc. Else, use emergency shutdown button situated on 1st, or 2nd floor.

4.4 Operation - Maintenance Actions

The maintenance operation is necessary once problem in column operation occurs, or there is an improvement which should be implemented. Due to column glass parts, it is necessary to be really careful, calmly follow planned procedure.

The lift truck can be used to get easier access to the column, and enables easier manipulation. To lower risk of column breakage, the possible threaten parts should be covered by padding material, such as bubble foil. All materials and tools used during maintenance action should be handled carefully, if possible stored in box to avoid these objects falling down to lower floors.

Lets consider maintenance action, which involves dismounting of one of the column sections. The maintenance manual consist of following steps:

- To dismount a section of the column it is necessary first to decrease the pressure caused by sections above the chosen one. It is done by tighten the spring hinges on flanges.
- The spring hinges on flanges on section's flanges are removed, the screws on flanges are symmetrically loosen, gradually until the flange is released. It is impossible to remove it, so it is left to rest on padding.
- Once, the section is released, it has to be held on place to avoid breakage.
- To enable complete removal of the section, it is often necessary to remove temperature measurement probes, which are connected by cables. It could be necessary to remove packing first.
- The removed glass part should be laid on padding to avoid breakage.

The set-up for installing the section back is basically reverse to dismounting. It is necessary to ensure that the pressure on individual screws in flanges is equal, as well as that the pressure on flanges (spring hangers) is even for whole column.

Practical Issues Connected to Operation of Kaibel Column

The operation of Kaibel column brings challenges, and practical issues, which need to be solved.

5.1 Leakage

Several individual leakages were observed during carrying out the experiments.

1. Loosen nut on the product tank level gauge – these level gauges are attached to reboiler, product tanks and feed tanks, once the nut by glass tube loosen, the liquid from tank leaks through.

Nuts should be checked regularly.

2. Leakage in between section 1 and upper Y section – vapor leaking from the column was condensing on the column. This leakage was due to teflon gasket, which is placed in between glass sections. Gasket was not sealing properly, because the cable of temperature sensor, which got stuck in between gasket and glass section.

The teflon gasket had to be replaced by new one.

3. Leakage from side stream 2 pipe withdrawal – vapor leaking and condensing. This glass part was already broken, due to tension glass broke again.

The pipe was glued to the proper place.

The column should be observed regularly during operation to find out possible leakages, the smell of the alcohols is also an indicator of problems.

5.2 Unreachable Temperature Profile

It was observed, that it could be unreachable to obtain steady temperature profile.

This was because of inequality of feed mixture. Methanol, and ethanol were missing due to leakage.

5.3 Feed Heater Temperature

The temperature of feed heater was increasing fast. This was because no feed flow was present, there was no feed mixture in the tank.

It is necessary to check the feed tank level regularly, and empty the product tanks before starting experiment.

5.4 Increased pressure drop in section between side streams

The unusual pressure drop behavior was observed by liquid seals on product side streams. See figure 5.1

The pressure difference at side stream 2 was increasing, while the pressure drop by side stream 1 was decreasing. Once the pressure drop (side stream 2) increases enough, the liquid seal was overcome, and the vapor leaked through product hose away. Once the overpressure was released, the pressure would start building up again. This flooding behavior is caused by high liquid hold up, but even after lowering the reboiler duty (and feed flow as well accordingly), the same phenomenon was observed even with relatively low reboiler duty/feed flow ratios.

Several explanations were proposed, and solutions implemented to solve this problem, these are listed below chronologically.

1. The pressure is increased due to vapor condensation in side stream pipe and coolers, and it is soaking more vapors from the column due to vapor condensation.

The geometry of coolers and liquid seals was changed – the liquid seals are now above coolers, the insulation for side stream pipes was added

2. Glass fragments in section internals, namely in product withdrawal device liquid collector above swinging funnel could prevent the vapor flow

The column was open, liquid poured through, no problem occurred.

3. Raschig rings interacting with structured packing, glass fragments inside structured packing increase the liquid holdup.

The column internals were checked, liquid was flushed through the section in opposite direction, Raschig rings and structured packing were split by wire sieve, new Raschig rings were inserted.

4. Pressure is building up in the other part of column, in prefractionator, vapor flow through split valve in prefractionator is stopped, and whole vapor flow goes to main column, which

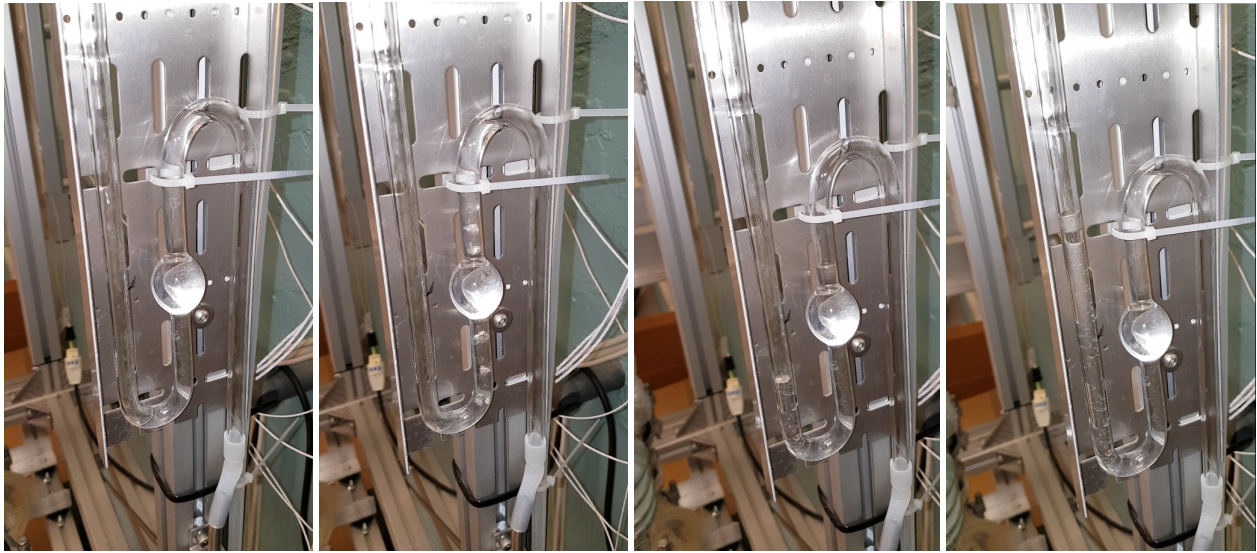


Figure 5.1: Pressure building up by product stream 2. The pressure gradually increases, once the liquid seal is overcome the vapor leak through. The pressure drop is then neglected. It starts building up again.

increases liquid hold up and causes flooding. The vapor flow is stopped due to accumulation of liquid in the valve. If the liquid can not go through downcomer, the accumulating liquid level would reach the cap and stop the vapor flow. Liquid accumulation can be caused by glass fragments or dirt in liquid downcomer. The schematic of liquid split valve is on figure 2.5.

The liquid seals were implemented above and below vapor split valves in both sections, see figure 5.2. The test experiments were carried to check the pressure differences. The highest pressure difference was observed in section below valve in prefractionator, the pressure difference is building up. When the liquid seal was overcome, the vapor starts leaking. This phenomenon occurs shortly after the feed to the column was turned on.

All explanations except last one, did not help to solve the problem. The last idea seems to explain the phenomenon quite well, yet the maintenance action has to take place, check the vapor split valves and clean the liquid downcomers.



Figure 5.2: Liquid seals for pressure measurement above (A) and below (B) vapour split valves in prefractionator (P) and main column (M)

Verification of Kaibel Column Performance

The experiments were carried to verify the overall performance of the column, and controller settings, as used by Dwivedi ().

6.1 Start-up

The figure 6.1 shows the heating up of the column during start up. The first temperature, which starts to increase is the temperature sensor closest to reboiler, then the sections are gradually heated up starting with section 7, then the vapour flow is split in prefractionator and main column, so the both branches are heated up simultaneously. Once the vapour reach the top of the column, section 3 is heated up, control of the distillate split valve could be turned on.

6.2 Open-loop Responses

Decentralized control structure consist of 4 temperature control loops. The settings of the PI controllers were verified, using open-loop step changes of manipulated variables. The liquid split valve was used to control temperature in prefractionator. The results of open loop step changes are presented in figure 6.2.

Using SIMC controller tuning rules for integrating processes proposed by Skogestad (). The tuning parameter, τ_c was used as proposed by Dwivedi (), for D and RL control loop equal to 1 min, while for RS1 and S2 equal to 2 min.

The obtained constant values agreed with the tunings presented in table 4.1.

6.3 Tuning of the Feed Heater Controller

The feed heater controller tuning was verified using open loop step response of manipulated variable, see figure 6.3

The tight and smooth controller tunings were again obtained using SIMC tuning rules. These values were compared with old tuning, see values in table 6.1.

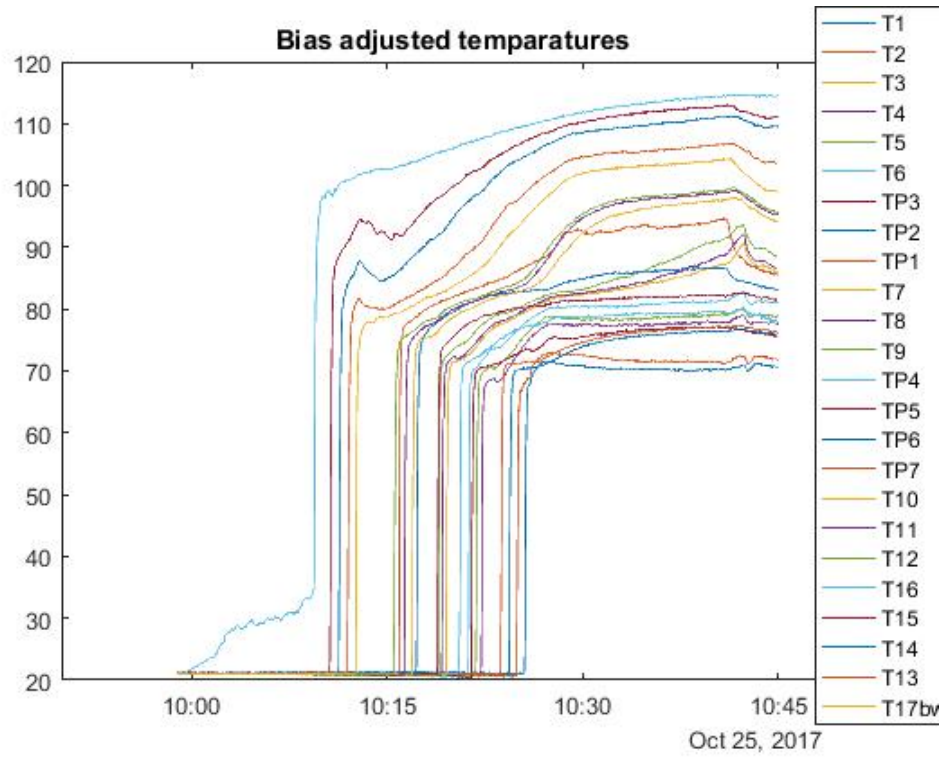


Figure 6.1: The heat up of the Kaibel column. The temperature profile of cold star-up.

Table 6.1: Feed heat controller tunings

Controller	Kp	I
tight	-0.052	2
smooth	-0.011	10
old	-0.03	10

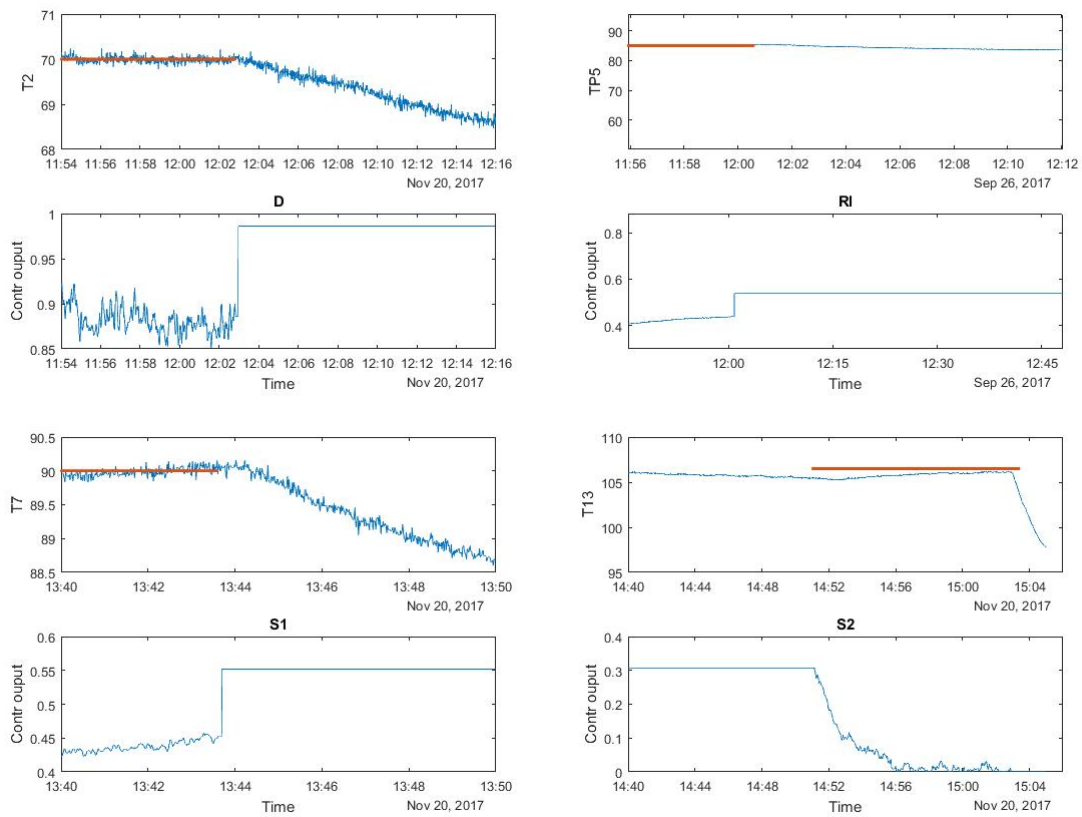


Figure 6.2: The open-loop step responses of 4 temperature control loops

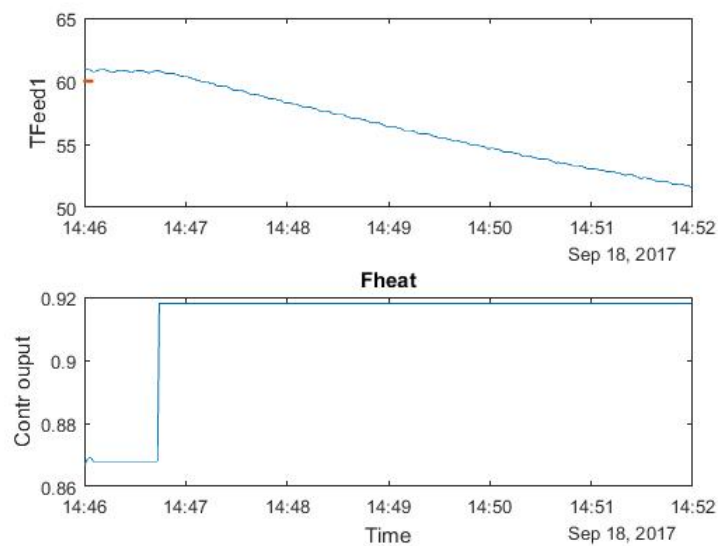


Figure 6.3: The open-loop step response of feed heat control loop.

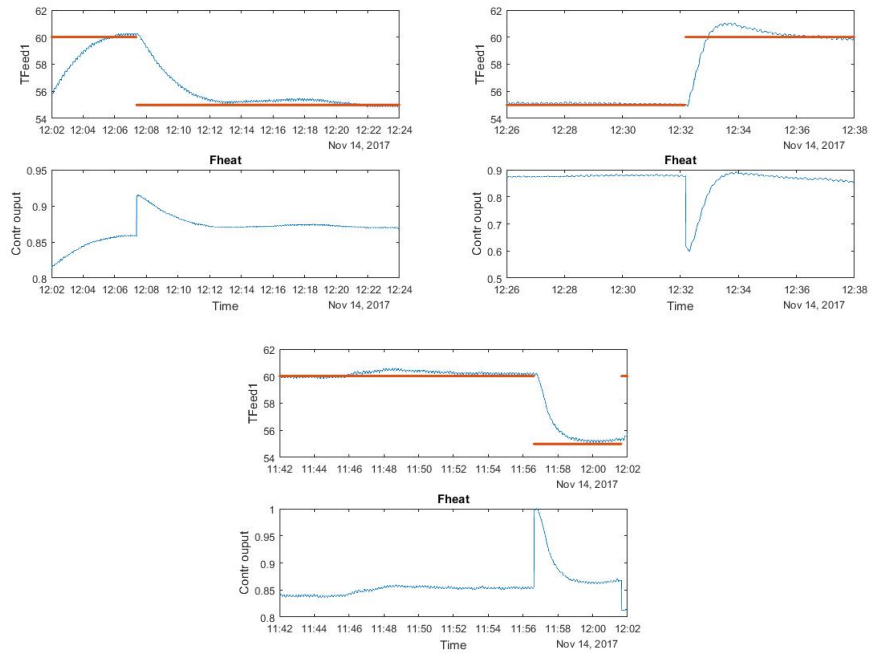


Figure 6.4: Feed heat controller. The closed-loop responses to setpoint change of 5°Cm and rejection of feed disturbance. Controller tunings from top: smooth, tight, old.

It was found, that old values of PI constants were chosen somewhere in between tight and smooth tuning- This values enables better performance than derived tuning after setpoint step change, or disturbance rejection. See figure 6.4

It should be mentioned, that the actual controlled temperature TFeed 1 is temperature measured on heating device. The temperature of the feed entering the column is lower due to time lag, and heat loss alongside the feed tube.

Measured feed heat temperatures are presented on figure 6.5. The TFeed 1 temperature is measured on heating device, the Tfeedheat is measured on feed tube near heater, and the TFeed 2 is measured on feed tube close to feed entering the column.

It would be problematic to control feed temperature directly and maintain safety operation, due to large delay. Possible solution would be installing cascade control, with feed temperature controlled by master controller, and TFeed temperature controlled by slave controller.

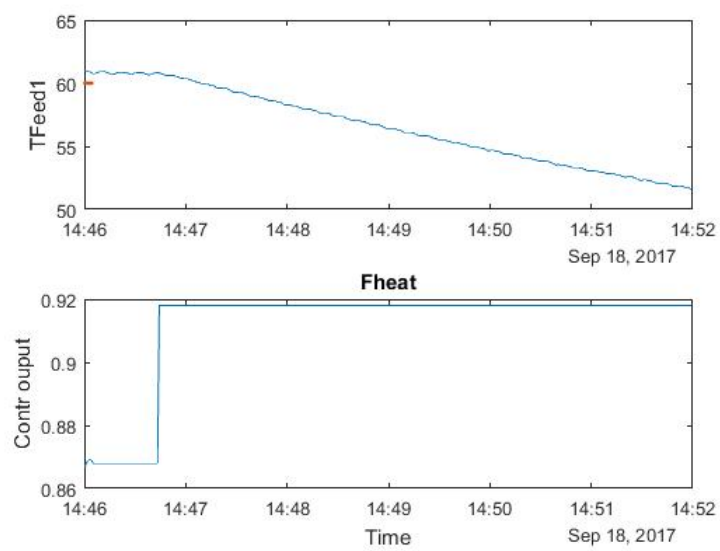


Figure 6.5: Measured feed heat temperatures
TFeed1 is measured on electric heater
Tfeedheat is measured on feedtube close to feed
TFeed 2 is measured on feed tube close to column feed

Conclusion

The main objectives were to carry out experiments on Kaibel column to verify its operation after recent rebuild, and shutdown, get familiar with operation of the column, and standardize the procedures.

The standard procedures for running the experimental Kaibel column were set, namely step-by-step manuals for start-up, shutdown, and maintenance actions. The evaluation of risks were executed, and enrolled into online Health Safety and Environment system at NTNU. The practical challenges met during operation of Kaibel column were described together with suggested solutions.

The experiments were carried out as a verification of performance. The steady state operation mode was successfully reached. The controller settings of 4 temperature control loops were verified, together with feed heat temperature control loop.

Unfortunately the verification experiments involving step changes of setpoint temperatures were not performed, due to operation problems, and challenges. The same deals for experiments involving vapor split valves.

Further Work

The further work would involve finishing the experiments verifying the performance. Step changes of setpoint temperatures for used control structure has to be performed. Later on, carry out experiments involving the vapor split valves as a manipulated variable.

The maintenance action should take place soon to verify the last theory explaining the problem with increasing pressure drop. Either to solve the problem, or to disprove the theory.

To look more into future, several improvements of the Kaibel column can be found.

The power switch of the feed pump can be connected to electrical circuit of whole column, so the shutdown button would turn off the energy input into the feed. Now, during automatic mode, the flow of the pump is stopped by setting the feed flow value to 0 after shutdown, but during manual mode, the pump would still work after shutdown.

The level controller on reboiler could be implemented, and used as a controlled variable, and the bottom product valve could work as a manipulated variable.

The pressure measurement could be added to ensure safety operation, the critical values would be called to operator, or even provoke automatic shutdown

The model predictive control of Kaibel column could be implemented, and compared to decentralized control structure.

Bibliography

Dwivedi, D. (2013). *Control and operation of dividing-wall columns with vapor split manipulation, Ph.D. thesis*. Norwegian University of Science and Technology, Department of Chemical Engineering, Trondheim, Norway.

Halvorsen, I. and Skogestad, S. (2006). Minimum energy for the four-product kaibelcolumn. aiche annual meeting.

Kaibel, G. (1987). Distillation columns with vertical partitions. *Chemical Engineering & Technology* 10, 1:92–98.

Kvernland, M. K. (2009). *Model predictive control of a Kaibel distillation column, Master thesis*. Norwegian University of Science and Technology, Department of Engineering Cybernetics, Trondheim, Norway.

Strandberg, J. (2011). *Optimal operation of dividing wall columns, Ph.D. thesis*. Norwegian University of Science and Technology, Department of Chemical Engineering, Trondheim, Norway.