

## STABILIZING OPERATION OF A 4-PRODUCT INTEGRATED KAIBEL COLUMN

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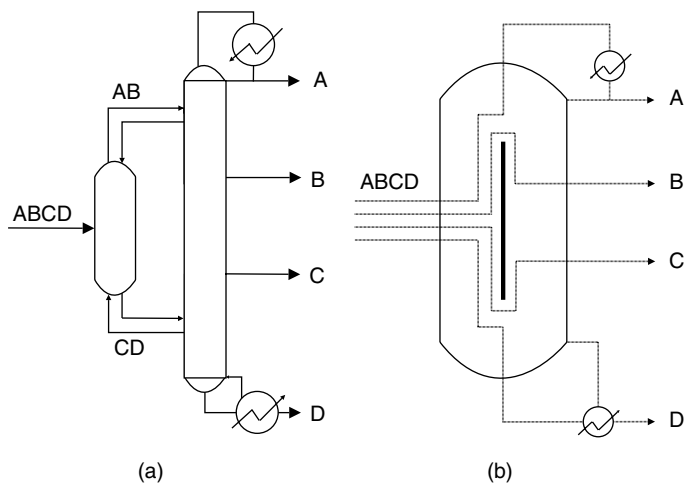
This paper considers the Kaibel column, a fully thermally coupled distillation column for the separation of four products in a single column with a single reboiler. The main reason for considering the Kaibel column is probably the potential capital savings compared to conventional arrangements with 3 columns in series. The authors of this paper have built a laboratory pilot plant of a Kaibel column with the purpose of investigating its operational performance and its control properties. We focus on minimizing the energy requirements to the column while keeping the products purities at their specifications. The possibility of manipulating the vapour flow on each side of the dividing wall to ensure optimal operation is also investigated.

KEYWORDS: distillation, divided wall, operation, thermally coupled columns

### INTRODUCTION

This paper considers the separation of four components in one fully thermally coupled column. The Kaibel column, introduced in 1987 (Kaibel, 1987) separates 4 products in a single column shell with a single reboiler. The main reason for considering the Kaibel column is probably the potential capital savings compared to conventional arrangements with 3 columns in series. The Kaibel column is an extension of the Petlyuk column (Petlyuk *et al.*, 1965). The Petlyuk column and the dividing wall column (DWC) (Wright, 1949) have been extensively investigated in the literature. Even though this research has shown potentially large savings in capital and operational costs, it has taken a long time for the industry to implement the ideas. However, the last 20 years have seen the technology come into use and there are now more than 40 divided wall columns in operation around the world (Adrian *et al.*, 2003). The Petlyuk arrangement can be extended to any number of products with the addition of vertical partitions or column shells. However, a practical realization of a 4-product Petlyuk column would be complex both in construction and operation. The Kaibel arrangement (Figure 1) is easier to implement because one would need only one vertical partition in a one-shell configuration. Both the Petlyuk and the Kaibel columns could be realized in a multi-shell arrangement, still retaining the energy-benefits, but one would of course lose the bonus of reduced capital cost as compared to the conventional three-column sequence. Instead of separating A/D in the prefractionator as in the Petlyuk arrangement, we here have a AB/CD split. This gives a somewhat higher energy requirement, but it is still has the potential to save energy as compared to a three-column sequence (Halvorsen, 2005). Especially, if the B/C separation is easy, the Kaibel configuration ought to be very

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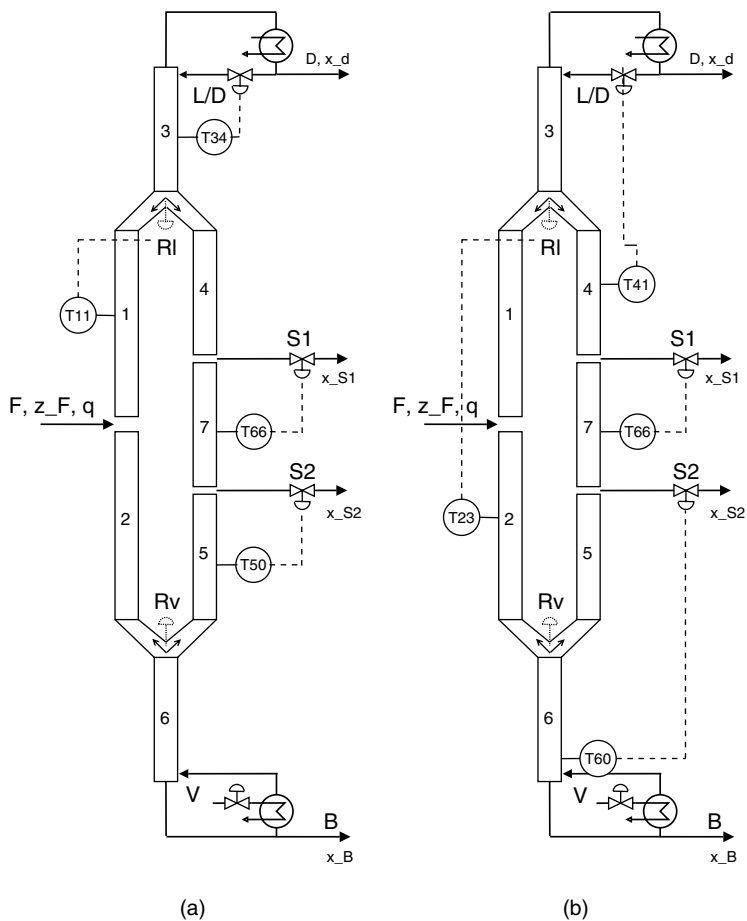
**Figure 1.** a) Kaibel column with prefractionator arrangement. b) Equivalent one-shell arrangement

competitive. BASF, who are the leading industrial company on the dividing wall technology, have at the moment the first Kaibel columns in operation (Kaibel *et al.*, 2004). However, to these author's knowledge there are not reported any lab-scale realizations of the column with thorough investigations of the operational and control properties of the column.

### PILOT PLANT AND COLUMN DATA

A pilot plant of the Kaibel column has been built at the Chemical Engineering Department, NTNU Trondheim. The column is put together by pre-fabricated column sections of internal diameter 50 mm. This leads, in effect, to a two-shell implementation (see Figure 2a), but it is equivalent to a divided wall column with no heat transfer across the partition wall. Two custom-made Y-pieces connect the parallel shells. The sections are made of vacuum-jacketed glass which offers very good heat insulation, and lengthwise sight-strips allow for inspection of the internals. The column is filled with glass Raschig rings for packing.

The reboiler is a kettle-type boiler of 3 kW capacity. The product streams (except bottoms) are controlled via solenoid operated swinging funnels built into the glass sections. Another funnel sets the liquid split,  $R_l$ , between the prefractionator and the main column. The column will initially be operated with a constant vapour split,  $R_v$ , however, two butterfly valves have been built into the column, one on either side of the partition for manual adjustment of the vapour flow. A total of 24 temperature sensors are distributed inside the column sections and make up the majority of measurements.



**Figure 2.** a) Stabilizing control scheme ( $c_3$ ) with four temperature loops (Valves are shown on individual streams for  $L/D$  and  $R_v$ , but in reality these are implemented as ratios using magnetically operated swinging funnels). b) Control scheme  $c_2$

The temperature measurements will be used in estimating the composition profile of the column and the purity of the products. Product samples will be analyzed with gas chromatography off-line to facilitate tuning and validation of the estimates.

The initial experiments will be run with a mixture of alcohols (methanol, ethanol, propanol and butanol), however, there are also plans to run the column with an alkane-mixture.

### MODELLING

The Kaibel column is modelled using a stage-by-stage model with the following simplifying assumptions: Constant pressure, equilibrium stages and constant molar flows. The vapour-liquid equilibrium is modelled using the Wilson equation. To model the column we have used 7 column sections with stages (see Figure 2a). Sections 1 and 2 make up the prefractionator, while the main column consists of sections 3-7. The prefractionator sections have 12 equilibrium stages, while sections 3-7 each have 8 equilibrium stages (our design necessitates the prefractionator having the same height as sections 4-7-5).

In this study, we separate the same four-component mixture that will initially be used in the pilot plant experiments (methanol, ethanol, propanol and butanol). We use an equimolar feed with partial preheating ( $q = 0.48$ ). We have specified 4 product purities:

$$\begin{bmatrix} x_D \\ x_{S1} \\ x_{S2} \\ x_B \end{bmatrix} \geq \begin{bmatrix} 0.975 \\ 0.94 \\ 0.94 \\ 0.975 \end{bmatrix}$$

The nominal operating point for the column have been found by optimization. The optimization criterion was to minimize the vapour boil-up,  $V$  (minimum energy input), with the model equations as equality constraints and the product purities as inequality constraints.

$$\begin{aligned} \min_{x,u} J &= V \\ \text{s.t.} & \\ f(\dot{x}, \dot{u}) &= 0 \\ h(x) &\leq 0 \end{aligned} \quad (1)$$

Data for the nominal optimum can be seen in Table 1. For the time being we decide to keep the vapour split  $R_v$  constant, at its optimal value.

**Table 1.** Nominal operating point. (Flows are scaled with regards to the feed.  $R_l$  and  $R_v$  are ratios)

Variable	Nominal value
$L$	2.7864
$V$	2.5107
$S1$	0.2437
$S2$	0.2530
$R_l$	0.3013
$R_v$	0.3233
$D$	0.2473
$B$	0.2560

### CONTROL OF KAIBEL COLUMN

The column has 7 dynamic degrees of freedom (valves):  $L$ ,  $V$ ,  $S1$ ,  $S2$ ,  $R_l$ ,  $D$  and  $B$ . In addition, it may be possible to adjust the vapour split,  $R_v$ , but this is not studied here. Column pressure is self-controlled at atmospheric pressure by the condenser design which has an open vent.

### STABILIZING CONTROL OF LEVELS

The condenser and reboiler holdups need to be controlled. We choose to use the “ $L/D$   $V$ -configuration” where the condenser level is controlled such that  $L/D$  remains as a degree of freedom in the top of the column and the reboiler level is controlled (using  $B$ ) such that  $V$  remains as a degree of freedom in the bottom of the column.

We are now left with 5 degrees of freedom ( $L/D$ ,  $V$ ,  $S1$ ,  $S2$ ,  $R_l$ ), and we need to use at least 4 of these to stabilize the column profile.

### STABILIZING CONTROL OF COLUMN PROFILE

In order to avoid “drift” in the column with undesirable breakthrough of impurities in the product we need to stabilize the column profile. First, in the prefractionator we need to maintain the split between components B and C. This may be done using  $R_l$  to control some temperature in the prefractionator, probably located in the top for good dynamic response. In the main column, we need to maintain the split between A and B in the top, B and C in the middle, and C and D in the bottom. This requires closing three additional temperature loops, for example using  $L/D$  to control a temperature in the top,  $S1$  to control a temperature in the middle section, and  $S2$  to control a temperature in the bottom (See Figure 2a).  $V$  and  $R_v$  remain unused. These loops need to be relatively fast and since composition measurements are usually slow or not available, we propose to use temperature loops, if possible with composition control in the outer cascade.

### LOCATION OF TEMPERATURE SENSORS

The objective of the inner loops is mainly to stabilize the column. But, in addition, we would like to keep the column reasonably close to its optimal operation, which is to minimize the energy usage ( $V$ ), while satisfying the four product purity constraints. One way of achieving this is the “self-optimizing control” approach (Skogestad, 2000). We will use parts of this approach to select the optimal location of the temperature measurements.

We here apply the minimum singular value method (Halvorsen *et al.*, 2003) for selecting the controlled variables. The procedure consists of the following steps.

1. Obtain a linear model  $G$  from the inputs  $u$  to the candidate controlled variables  $y$ .
2. Scale the inputs  $u$  such that the effect of each input is the same on the objective function.
3. Obtain the scaled gain  $G_s$  of the model by scaling the outputs using sum of their optimal range and their implementation error (“span”).
4. Select controlled variables that maximize the minimum singular value  $\sigma$  of the scaled gain matrix  $G_s$  from  $u$  to  $y$ .

In the following we will assume that we have an on-line measurement of the bottoms product composition but that the other product streams do not have this feature. The available measurements (candidate controlled variables) are then the temperature at each stage and the bottoms composition,  $x_B$ . We also include our 5 inputs as possible outputs in the analysis. We then have a total of 71 candidate variables from which we want to find the best set  $c$  of 5 variables to keep at constant set-points.

$$y = \{L/D, V, S1, S2, R1, T_1 \dots T_{65}, x_B\} \quad (2)$$

The implementation errors used to obtain the scaled matrix were: 10% for flow measurements, 0.5 K for temperatures and 0.001 for the mole fraction. Using the exact branch and bound method (Cao *et al.*, 1997) we find that we should pick the following five variables:

$$c_1 = [T_{24} \quad T_{41} \quad T_{56} \quad T_{68} \quad x_B] \quad (3)$$

These five give a minimum singular value  $\sigma = 113.53$ . We see that the method chooses  $x_B$ , which one would expect to be a good variable. Now, we will investigate the strategy where we have constant vapour boil-up (we pick  $V$  as both input and output) and look for the the best 4 remaining outputs. We then get the following controlled variables:.

$$c_2 = [V \quad T_{23} \quad T_{41} \quad T_{60} \quad T_{66}] \quad (4)$$

The location of the measurements are visualized in Figure 2b. We see that the method does not choose the same outputs as before, even though some of them are retained. Here the minimum singular value  $\sigma$  is reduced to 1.5265, indicating that this it is not an optimal strategy to keep  $V$  constant.

If we now go back to our preliminary control structure seen in Figure 2a, we can evaluate the minimum singular value for choosing these outputs:

$$c_3 = [V \quad T_{11} \quad T_{34} \quad T_{50} \quad T_{66}] \quad (5)$$

This gives  $\sigma = 1.200$  which is somewhat lower than for  $c_2$ .

### LOSS CALCULATIONS

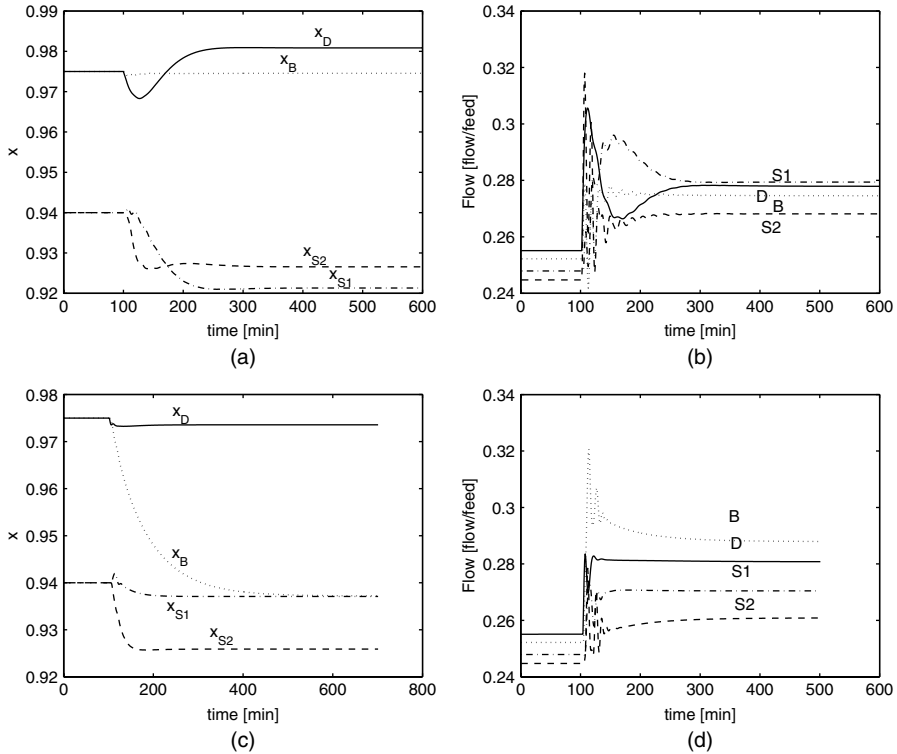
In Table 2 we show the percentage loss in the cost function when disturbances are introduced. We keep the sets ( $c_2$  and  $c_3$ ) of temperatures at their optimal values and increase  $V$  to reach feasibility with respect to the purity constraints. We see that  $c_2$  does indeed produce smaller losses than  $c_3$  as indicated by the singular value.

### DYNAMIC SIMULATIONS

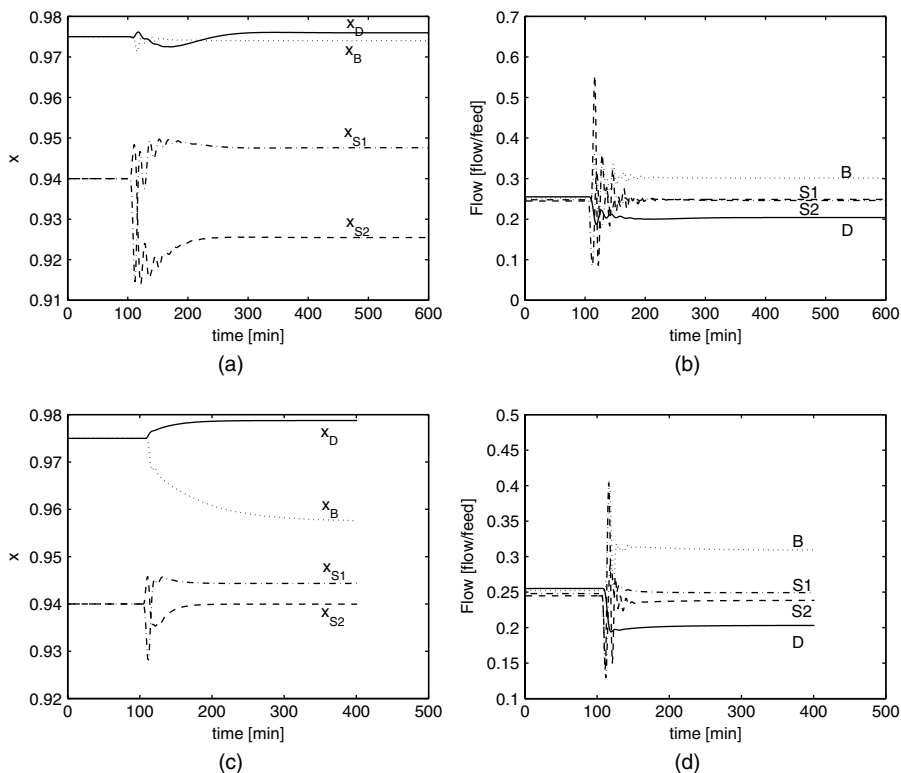
The two control schemes,  $c_2$  and  $c_3$  were simulated to check the dynamic responses. In each case the four temperature loops were implemented using PI-controllers tuned with Skogestad's IMC tuning rules (Skogestad, 2003). Figure 3 shows the responses from a 10% increase in the feed rate. We note that scheme  $c_2$  does better with regards to the

**Table 2.** Loss in cost function due to disturbances when temperature loops are applied

Disturbance	$c_2$	$c_3$
$F: 1.0 \rightarrow 1.1$	0.39%	0.39%
$z_A: 0.25 \rightarrow 0.20$	12.0%	26.2%
$z_B: 0.25 \rightarrow 0.20$	3.32%	10.5%
$z_C: 0.25 \rightarrow 0.20$	0.12%	1.04%
$z_D: 0.25 \rightarrow 0.20$	0.03%	2.10%



**Figure 3.** Disturbance response. 10% increase in feed rate. Top: response for controlled set  $c_2$ . Bottom: response for controlled set  $c_3$



**Figure 4.** Disturbance responses.  $z_A = 0.20$ ,  $z_D = 0.30$ . Top: response for controlled set  $c_2$ . Bottom: response for controlled set  $c_3$

bottoms composition, while  $c_3$  has a somewhat better response in terms of the sidestream compositions. In Figure 4 the feed composition of component A, ( $z_A$ ), has been decreased from 0.25 to 0.20, while  $z_D$  is increased from 0.25 to 0.30. Again we see that  $c_3$  keeps the sidestream compositions better than  $c_2$ , but the bottom composition drifts relatively far away with the set  $c_3$  as compared to the set  $c_2$  found by the singular value method. To get back to the specified purities we can adjust the boil-up  $V$ .

## EXPERIMENTAL

The initial runs on the rig showed that there were very small amounts of vapour actually reaching the top of the column. Increasing the heat input to the reboiler only caused flooding problems in the lower end of the column, especially evident in the lower junction. It



was clear that the tall column was experiencing greater heat losses than allowed for proper operation. As the glass sections themselves are very well insulated, the main culprit was identified as the two butterfly valves for adjusting the vapour split. Even though they were insulated on the outside, it was decided to install additional heating to compensate for the losses. A heating tape of 440 W was installed on each valve with new insulation on the outside. The heating tapes are controlled to keep the wall temperature at about 100 °C. A third heating tape was installed on the feedpipe for pre-heating the feed. This will not only help in getting more vapour to the top of the column, but also provides the feed temperature as an additional variable for manipulation.

Detailed experimental results will be provided at the conference presentation.

## CONCLUSIONS

In this paper we have discussed the Kaibel distillation column for the separation 4 products. The Kaibel column is interesting because it has the potential for large capital investment savings as well as reduced energy consumption when compared to conventional distillation sequences. To stabilize the column operation one has to close a total of 4 control loops. The remaining degrees of freedom (in our case  $V$  and potentially  $R_V$ ) can be used to ensure optimal operation according to some economic objective.

The minimum singular value method has been applied to the problem of finding the optimal location of temperature measurements for stabilizing control. The resulting control scheme has been compared with a predefined scheme using both steady-state and dynamic simulations, showing that the method can be useful in selecting measurements.

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