CHEMICAL ENGINEERING TRANSACTIONS
VOL. 69, 2018
A publication of AIDIC
The Italian Association of Chemical Engineering
Online at www.aidic.it/cet
Guest Editors: Elisabetta Brunazzi, Eva Sorensen

ISBN 978-88-95608-66-2; **ISSN** 2283-9216

New Process Control Concepts for Energy Efficient Operation of Reactive Dividing Wall Columns

Lisa S. Egger*, Georg Fieg

Hamburg University of Technology, Institute of Process and Plant Engineering
Am Schwarzenberg-Campus 4, 21073 Hamburg, Germany
lisa.egger@tuhh.de

In this study a concentration control concept is employed for energy efficient operation of a reactive dividing wall column. The control concept takes into account the liquid split as a manipulated variable in order to manipulate the component flow in the column, while the heavy key reactant concentration on the top stage in the prefractionator serves as control variable. The dynamic performance as well as the ability to adjust energy minimal steady states after disturbances is analysed and evaluated by comparison of the proposed concept to one with a fixed liquid split.

1. Introduction

Process integration is an important method to increase process efficiency in chemical industry. A highly integrated process is represented by the reactive dividing wall column (RDWC), which combines a chemical reaction and several separation steps in one column shell. Theoretical studies state that the energy demand of RDWCs can be reduced up to 15 % and investment costs up to 30 % in comparison to conventional separation sequences (Schröder and Fieg, 2016). These savings offer great potential both economically as well as ecologically. However, the high degree of integration also leads to complex process dynamics and phenomena like multiple steady states, which makes it difficult to operate RDWCs. Ensuring constant product purities while maintaining the energy efficiency in cases of disturbances places high demands on the development of a control system.
In order to finally introduce the RDWC into industrial application it is necessary for the process to outperform other alternatives, like the reactive distillation sequence or conventional reactor and distillation sequences. Therefore, this paper aims to contribute to the research about energy efficient control strategies for the RDWC, which is a crucial aspect in the continuous operation of this apparatus.
In case of decentralized control, typical control variables for columns are temperatures or concentrations. While temperature control shows fast dynamics, due to the direct measurement of the controlled variable, it is only the representative of the actual target value in the column. Because of the strong interactions between reaction and separation, as they are likely to occur in RDWCs, the concentration in the column might not be uniquely represented by the temperature. This can lead to deviations of the target value. Concentration control on the other hand, has the drawback of slow dynamics, especially when controlling the product concentrations in the outlet streams of the column. However, due to the direct measurement of the target value, it is possible to control concentrations very precisely. In order to focus on the energy efficient control and the underlying phenomena without further overlapping effects that arise from temperature control, concentration control will be applied in this work.

1.1 Control of Reactive Dividing Wall Columns

The development of control strategies for the RDWC is impeded by the highly nonlinear nature of the process. To date a few simulation studies about possible control structures for different applications and reaction systems have been carried out. Mostly decentralized concepts are studied, but also first results on model predictive control are available. Weinfeld et. al (2018) give a comprehensive review on recent developments of control strategies for the RDWC. Egger and Fieg (2017) also present first experimental results on the control

of a RDWC pilot plant. Despite the recently active research about control strategies no control concepts for energy efficient control of RDWC are presented yet.

1.2 Energy Efficient Operation and Control

Theoretical studies prove the high potential regarding energy efficiency of the RDWC. Schröder and Fieg (2016) show that the component flow in the column has a major effect on the energy demand of the column. It is known that, for a suitable reaction system, the high boiling component should always distillate below and the light boiling component above the partition wall. Middle boiling components should be distributed on both ends of the partition wall, according to the preferred component split of the operation point. A variable that has a great impact on the component flow in the column is the liquid split. The liquid split controls the distribution of the reflux flow to both sides of the partition wall. Changing the liquid split affects the distribution of the components in the prefractionator and the main column. However, to date no control concept for the RDWC is known which utilizes the liquid split as a manipulated variable to assure energy efficient operation. In contrast, the liquid split has been investigated and proposed as control variable for non-reactive dividing wall columns (Halvorsen and Skogestad, 1997). A comprehensive overview about applied control structures, including energy efficient control for dividing wall columns, can be found at Yildirim et al. (2011). Ling and Luyben (2009) propose a control structure for the dividing wall column where the liquid split controls the heavy boiling component concentration at the top of the prefractionator. This approach shall obtain the correct component flows in the column by preventing an accumulation of the heavy boiling component above the partition wall. In the following this approach will be transferred to the reactive dividing wall column.

2. Methodology

This study is conducted utilizing a steady state and dynamic RDWC model. In order to evaluate the energy efficient control of the RDWC, two aspects will be considered. First sensitivity analyses are conducted to identify the energy minimal liquid split for each applied disturbance case. Second, two control concepts are analysed, whereas one concept adjusts the liquid split as manipulated variable according to a measured concentration in the column, while the other concept serves as reference concept and will be operated with a constant liquid split.

2.1 Simulation Model

The employed comprehensive RDWC model is developed at the Institute of Process and Plant Design at the Hamburg University of Technology and was extensively validated by experimental results (Egger and Fieg 2018). To solve the model equations Aspen Custom Modeler is employed. A modular modelling approach is selected, to allow simulating different column scales and configurations. The configuration applied in this work consists of 6 column section models and 5 collector/distributor models. Additionally, models for the distillate vessel, reboiler and condenser are included. The component separation is calculated using the well-known equilibrium stage approach. For the calculation of the pure component vapour pressure the Antoine equation and for the binary phase equilibria the UNIQUAC equations are employed. The reaction is included in the MESH equations, as shown exemplary in Eq. (1).

$$\frac{dNx_i}{dt} = \mathrm{L}_{in}x_{in,i} + \mathrm{V}_{in}y_{in,i} - \mathrm{L}_{out}x_{out,i} - V_{out}y_{out,i} + m_{cat}\sum_{j=1}^{n_R} r_{i,j} \tag{1}$$

The applied reference reaction is the transesterification of 1-butanol and n-hexyl acetate (Eq. 2). It is assumed that the reaction takes place in the liquid phase only. A second order power law approach is chosen to describe the reaction kinetics for the selected reaction system.

$$1-butanol \; + \; n-hexyl\; acetate \; \rightleftharpoons \; 1-hexanol \; + n-butyl\; acetate \tag{2}$$

For the phase equilibria and reactions kinetics, the property parameters from Egger and Fieg (2018) are employed. Additionally, pressure drop and heat losses to the surroundings are calculated. A self-adjusting vapour split allows taking into account the dynamic effects of changes to the columns hydrodynamics.

2.2 Energy Minimal Operation at Disturbances

In case of disturbances, the liquid split has to be adapted to assure the proper component distribution in the column and thus the energy efficient operation. To identify the optimal liquid split values, for each disturbance case sensitivity analyses are conducted. In this work, disturbances in feed flow and feed composition are investigated (shown in table 1). The specific disturbance variable is changed and a steady state simulation is conducted. Afterwards the liquid split is varied and the corresponding heat duty at each new steady state is

examined. The results of the analyses are shown in figure 1. The lower boundary for the liquid split is set to 0.2, as previous investigations showed that smaller values might lead to unfeasible internal flows, for example maldistribution or vapour channeling, in the column. For values greater than 0.6 the energy demand rises monotonously and is therefore not shown. Each case holds at least one minimum in the presented range. In two cases (feed flow and feed concentration increase) a second local minimum occurs. In these cases the liquid split could settle into two different states, depending on the initial state and the direction of the disturbance. The minimal heat duties and the corresponding values for the liquid split can be found in table 1. The analyses show that the liquid split has to be adjusted in a range from 0.2 to 0.38 in order to meet the minimal energy demand in case of all four disturbances.

Table 1: Investigated disturbance cases with corresponding minimal heat duty and liquid split

Case	Disturbance variable	Intensity	1st minimum		2nd minimum	
			Q min [kw]	liquid split [-]	Q min [kw]	liquid split
1	Feed flow	- 10%	0.753	0.25		
2	Feed flow	+ 10%	1.113	0.3	1.241	0.2
3	hexyl acetate in feed flow	- 10 mass%	0.715	0.2		
4	hexyl acetate in feed flow	+10 mass%	1.209	0.2	1.238	0.38

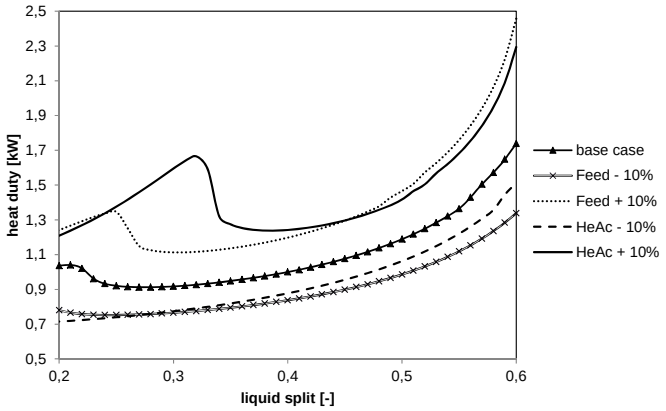

Figure 1: Identification of the minimal heat duty and corresponding liquid split in case of disturbances

2.3 Concentration Control Structures

In this study two decentralized concentration control structures will be analysed and compared. As mentioned before, concentration control has the major advantage to assure precise control of the actual target values. Therefore, both control structures contain three concentration control loops to control the key components in each product stream (butyl acetate in the distillate stream, butanol in the side stream and hexanol in the bottom stream). Two level control loops are implemented to maintain the distillate tank and reboiler level. Control structure 1 (CS1) possesses an additional control loop, which controls the concentration on the top tray in the prefractionator by manipulating the liquid split (figure 2). By controlling the heavy key reactant concentration a stable conversion is assured while at the same time the accumulation of hexyl acetate and hexanol in the top of the column is prevented. In order to achieve the identified energy minimal operation points at disturbances (table 1) the set point for the liquid split control loop will be adjusted. Therefore, the optimal values for the hexyl acetate concentration on the controlled tray are taken from the former sensitivity analyses. Control structure 2 (CS2) serves as reference case and is operated with a constant liquid split. The tuning procedure for the concentration control loops was carried out using the relay feedback test and Tyreus-Luyben tuning parameters (Tyreus and Luyben 1992). In order to enable an equitable comparison of CS1 and CS2 the same tuning parameters were applied. The applied control parameters are listed in table 2.

Table 2: Tuning parameters of all control loops in CS1 and CS2

Controller	QC1	QC2	QC3	QC4	LC1	LC2
K_p [%/%]	12.8	8	121.6	0.73	28.6	133.3
T_i [min]	145.2	66	118.8	39.6	-	-

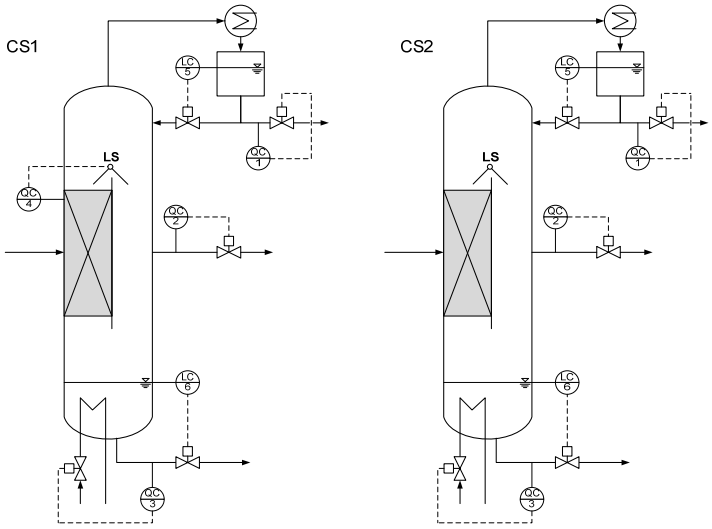

Figure 2: Control structures CS1 (left) with liquid split control loop and CS2 (right) with fixed liquid split

3. Results

The dynamic behaviour of the control structures 1 and 2 is analysed and compared for all disturbance cases shown in table 1. First the dynamic response of the product qualities will be shown in order to compare the performance of the two structures. In a second step the heat demand for each case will be analysed in detail. Therefore the minimal heat demand and the corresponding liquid split will be compared to the results of both control structures.

3.1 Dynamic Performance of CS1 and CS2

In figure 3 the dynamic courses of the main product components are shown for disturbances in feed flow and feed concentration. The red lines represent CS1 while the black lines represent CS2 with fixed liquid split.
Feed flow decreases and increases, as shown in figure 3a) and 3b), can be adjusted by both control structures in about 5 h. All product concentrations in the readjusted steady states can be maintained in specification. However the deviations during the settling time differ among the different product streams. The product obtained at the reboiler stream only shows small deviations of max. 0.3 mass%. Also the butyl acetate concentration in the distillate stream is effectively controlled and shows maximal deviations of 0.5 mass% during settling time. In contrast, the feed flow disturbances have a stronger influence the side stream concentrations. The occurring deviations of the butanol concentration in the side stream show maximum values of 4 mass% for CS1 and 3.3 mass% for CS2 in case of feed flow reduction. For a feed flow increase the deviation is about 2.5 mass% for both control structures. Comparing both structures shows a very similar performance. The only difference is the course of the concentrations during the settling time. While CS2 shows a very smooth change of the concentrations, CS1 with adjustable liquid split, shows oscillations, especially in the side stream concentration. The varying liquid split, which responses to the disturbance as well as to the simultaneous set point change, interacts with the other control loops and strongly affects the concentration profile. This effect can be observed especially in the side stream, as the liquid split directly influences the component flow on both sides of the partition wall.
Figure 3c) and 3d) show the control response in case of a change of the hexyl acetate concentration in the feed flow by +/-10 mass%. Here again the settling time for both structures is similar at about 7 h and deviations in the reboiler and distillate concentrations are small, approx. 0.2 mass% for hexanol and 0.5 mass% for butyl acetate. The side stream concentration shows larger deviations, approx. 3 mass% for CS1 in case of hexyl acetate decrease and 2 mass% in all other cases. However, it is noticeable, that the side stream concentration of CS2 in the case of a hexyl acetate decrease, shows oscillations and is not reaching a steady state at the end of the simulation time. CS1 with variable liquid split reaches a steady state concentration and maintains the product specifications.

3.2 Energy Efficient Control

The examination of the dynamic performance does not show significant differences between CS1 and CS2 regarding settling time and steady state concentrations. In order to further analyse the readapted steady states the heat demand and the corresponding liquid split values for both structures and all disturbance cases

are investigated. Figure 4a) shows the heat demand for both control structures which arise in dynamic simulations as well as the minimal heat demand identified during steady state sensitivity analyses (as shown in table 1). In case of a feed flow increase, the adjusted heat duty of both control structures is similar and very close to the minimal heat duty. Corresponding to that the liquid split values, shown in figure 4b, are also very similar, indicating that both control structures achieved the same operation point. In case of a feed flow reduction, the heat demand of CS2 is approx. 2 % greater than the minimal hat duty. Interestingly, the corresponding liquid splits display different values. CS1 adjusts the liquid split successfully to 0.25, while in CS2 the liquid split is constant at 0.3. However, in this case, as can be seen in figure 1, the heat duty is not very sensitive to the liquid split in a range from 0.2 and 0.35. Therefore, the same heat duty can be achieved with different liquid split values. In case of feed concentration disturbances, the heat demand differs more significantly. While the heat demand of CS2 exceeds the minimal value by 33 % in case of a hexyl acetate increase, CS1 can settle almost exactly to the energy minimal operation point. In case of a hexyl acetate reduction, the heat demand of CS1 again fits the minimal heat duty, while CS2 requires 8 % more energy. These findings are supported by the resulting liquid split values. CS1 adjusts the liquid split in both cases close to the optimal values while the constant liquid split deviates up to 50 % from this value. The results show that the adjustment of the liquid split is useful to maintain minimal heat duty during operation and thus achieve savings in operation costs. However, the extent of the savings is dependent on the sensitivity of the minimal heat duty towards the different disturbance cases, as can be seen in the sensitivity analyses in figure 1.

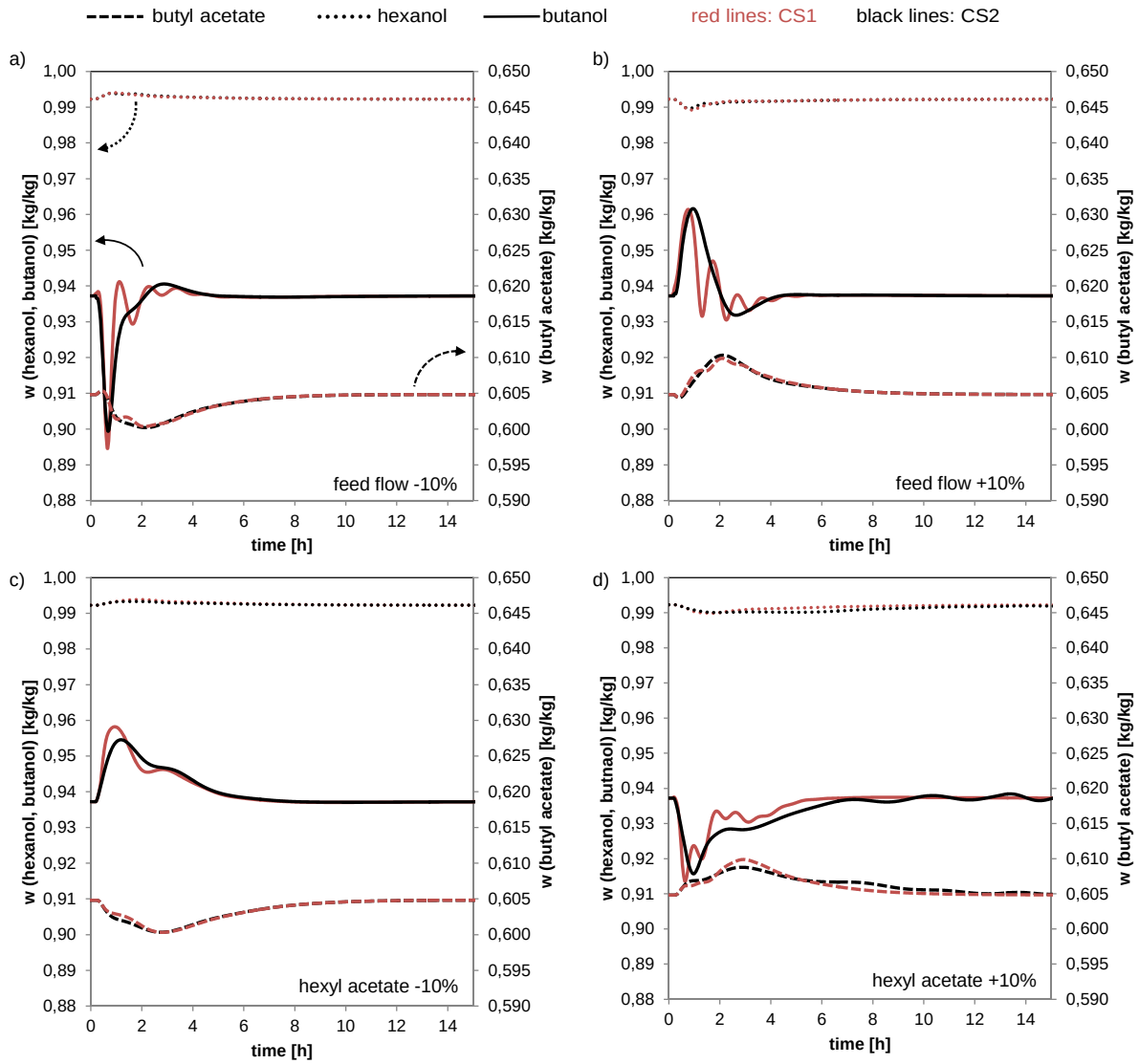

Figure 3: Dynamic course of the three main components in the product streams (distillate stream: butyl acetate, bottom stream: hexanol, side stream: butanol) after feed flow disturbances by -10 % (a) and +10 % (b) and feed concentration disturbances by -10 % hexyl acetate (c) and +10 % hexyl acetate (d)

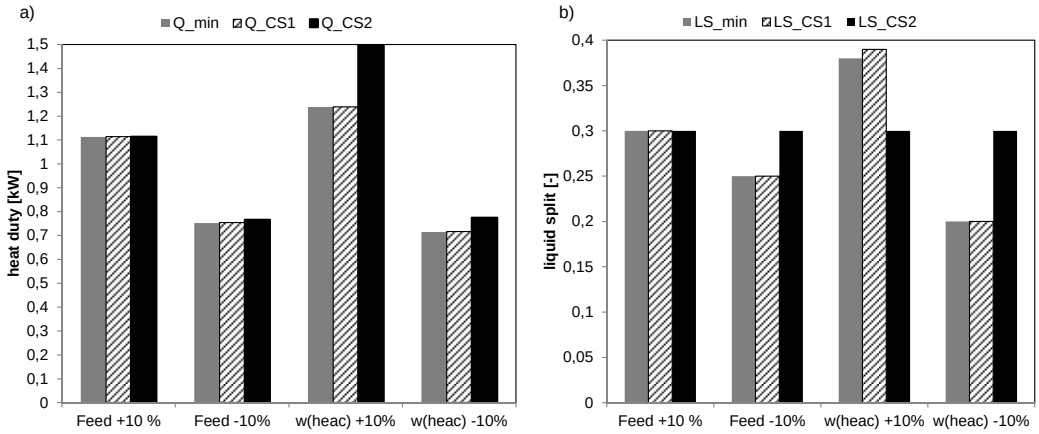

Figure 4: Comparison of minimal heat duties and liquid split values for the investigated disturbance cases

4. Conclusions

In this paper a control structure for the energy efficient control of a RDWC is suggested. Therefore, the heavy key reactant concentration on the top stage in the prefractionator is controlled by the liquid split. In addition a set point adaption is employed to meet the energy minimal concentration, which was identified in sensitivity analyses. All examined disturbances, changes in feed flow and the feed concentration, can be successfully adjusted by the control structure while the specifications of the product streams can be maintained precisely. A comparison with a control structure with fixed liquid split shows that significant reduction in the heat duty can be achieved for the readjusted steady states while the extent of the saving is dependent on the specific disturbance. While the proposed structure always reaches the energy minimal operation point, the heat demand of the structure with fixed liquid split is up to 33 % higher that the minimal value. Comparing the performance of both structures, with and without liquid split control loop, proofs that the energy efficient liquid split-adjusting control structure has no disadvantages concerning settling time and accuracy. This work shows that it is possible to operate RDWCs energy efficient even in the event of disturbances, which is an important step towards the application of the RDWC into industrial practice.
After proving the feasibility of the energy efficient control of RDWCs, in a next step the concept will be extended to temperature control in order to improve the dynamics of the system.

References

Egger L.S., Fieg G., 2017, Decentralized process control of reactive dividing wall columns, Chemical Engineering Transactions, 57, 1693-1698

Egger T., Fieg G., 2018, Dynamic process behaviour and model validation of reactive dividing wall columns, Chemical Engineering Science, 179, 284-295

Halvorsen I.J., Skogestad S., 1997, Optimizing control of Petlyuk distillation: Understanding the steady-state behaviour, Computers & Chemical Engineering, 21, 249–254

Ling H., Luyben W.L., 2009, New control structure for divided-wall columns, Industrial & Engineering Chemistry Research, 48 (13), 6034–6049

Schröder M., Fieg G., 2016, Influence of Reaction System Properties on the Energy Saving Potential of the Reactive Dividing-Wall Column: Separation Properties, Chemical Engineering Technology, 39 (12), 2265–2272

Tyreus B.D., Luyben, W.L., 1992, Tuning PI controllers for integrator/dead time processes, Industrial & Engineering Chemistry Research, 31 (11), 2625–2628

Weinfeld J.A., Owens S.A., Eldrige R.B., 2018, Reactive dividing wall columns: A comprehensive review, Chemical Engineering & Processing: Process Intensification, 123, 20–33

Yildirim Ö., Kiss A.A., Kenig E.Y., 2011, Dividing wall columns in chemical process industry. A review on current activities. Separation and Purification Technologies, 80 (3), 403–417