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Decentralized Process Control of Reactive Dividing Wall Columns

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In this work a decentralized control structure for the reactive dividing wall column (RDWC) is proposed. Based on sensitivity analyses with a steady state model the optimal pairing of controlled and manipulated variables is shown. For the first time an experimental study about a RDWC control structure is conducted. Therefore, an enzymatic catalysed reactive dividing wall column in pilot scale is employed. Several experiments are carried out systematically in order to examine the dynamic performance under the influence of disturbances. In this paper the results of disturbances in heat duty and set point changes are shown and discussed.

1. Introduction

The chemical industry is strongly influenced by the increasing demand for efficiency, ecologically as well as economically. In order to meet these demands, process integration has become a very important method in process industry. By combining different unit operations in one apparatus investment costs are reduced and energy efficiency is improved. A highly integrated process is represented in this work by the reactive dividing wall column (RDWC). This apparatus combines a chemical reaction and several separation steps in one column shell. At the same time up to four high purity products can be obtained. Due to the high complexity of this apparatus chemical industry is reluctant to build and operate RDWCs. To date various papers have been published about design and steady state operation of RDWCs, for example by Barroso- Muñoz et al. (2009) or Bumbec et al. (2009). However, there are only few papers about the control and dynamic operation. Experimental studies about the controlled performance of RDWCs are still pending.

This work aims to contribute to the research about control of RDWCs. For the first time, a comprehensive experimental study is conducted in order to gain insights in the dynamic performance of a real RDWC pilot plant. In a first step the control structure is presented and described. Sensitivity analyses are conducted aiming to identify suited sensor positions for the temperature control. With the knowledge of the best fitting positions a RDWC pilot plant is operated. Several operating points are analysed in a total of six experiments. The control structure is tested by applying temporary and continuous disturbances in heat duty and feed flow as well as by set point changes.

1.1 Control strategies for the reactive dividing wall column

In contrast to control of reactive distillation and dividing wall columns, there are only little publications about the control of the next integration step, the RDWC. All of the studies to date are theoretical, simulation based approaches. Transesterification with a chemical catalyst is the most examined reaction regarding control of RDWCs. For this reaction several decentralized control strategies are proposed for example by Ignat and Kiss (2013), Wang et al. (2011), Li et al. (2016), Zheng et al. (2016) and Hernández et al. (2009). Besides decentralized concepts Qian et al. (2016) and Rodríguez et al. (2016) propose model predictive control strategies for the RDWC.

2. Decentralized temperature control of the RDWC

In this work a decentralized control structure for the RDWC is proposed and analysed. A steady state model is employed to identify suited positions for the temperature sensors of the applied temperature control loops.

The control structure is shown in *Figure 1*. In the column the level of the distillate vessel as well as the bottom-level have to be controlled in order to avoid draining or flooding of the tank and the column respectively. Therefore, two level control loops are employed. The distillate vessel level is controlled by the distillate stream while the bottom stream serves as manipulated variable for the bottom level. For these level control loops proportional controllers are applied. The product specifications are maintained by a two-point-temperature control structure. Usually composition control is connected with large dead times. Therefore, temperature profiles can serve as representatives for composition profiles in the column. This allows a direct online measuring and thus fast control responses. In this work two temperatures, one in the prefractionator and one in the main column, are chosen as controlled variables. They enable the control of the distillate and the side stream compositions. For these two temperature control loops PI controllers are applied. In this study the heat duty is set to a fixed value for technical reasons and is currently not employed as a manipulated variable. The required value for the heat duty is calculated based on simulation studies and previous findings from experimental runs. This value has to be chosen carefully in order to meet the product specifications even under the influence of disturbances. Finally, a pressure control loop ensures a steady operating pressure at the top of the column.

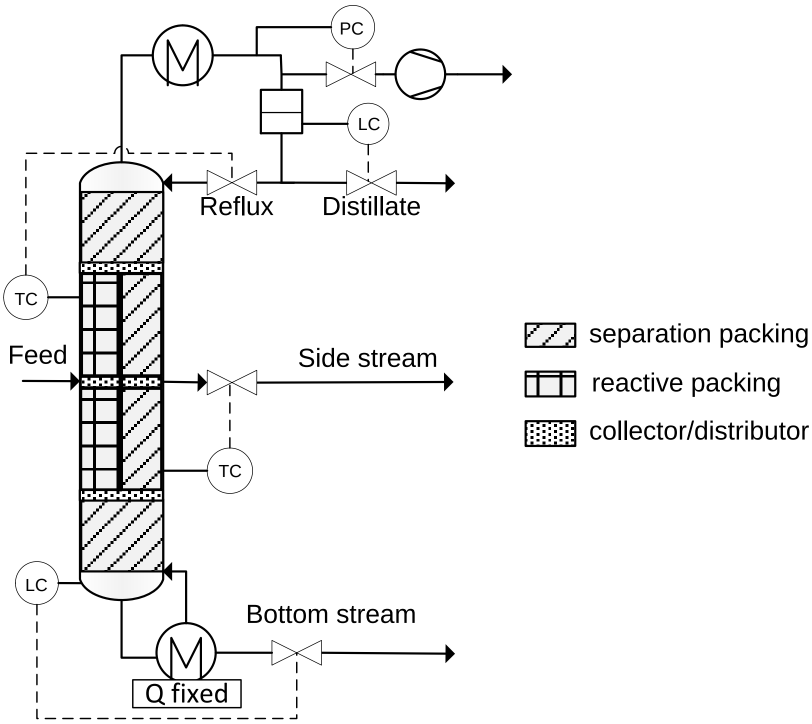

Figure 1: Two-point-temperature control structure for the RDWC with fixed heat duty (Q)

2.1 Simulation model

A series of sensitivity analyses are conducted using a steady state model of a RDWC in order to identify suited positions for the temperature sensors. The employed model was developed at the Insititute of Process and Plant Engineering and provides a high reliability due to extensive experimental validation (Egger, Fieg 2016). The steady state model is based on the equilibrium stage approach. It is implemented in the software Aspen Custom Modeler. The model structure is divided into a main model for the column and several models for the periphery, such as the distillate vessel, the reboiler and the condenser. In the main model the column is divided into column sections, which contain stages in the reactive zone and the separation zone, and collector/distributors between these sections. Specifications of the packings as well as for the catalyst can be adjusted according to the requirements. The separation performance is calculated by the well-known MESH-equations.

In this work the model was used in a configuration that represents the RDWC pilot plant at the Hamburg University of Technology. Therefore, the reaction model was adapted to the employed reference system, the transesterification of n-butyl acetate and 1-hexanol forming 1-butanol and n-hexyl acetate. Immobilized Candida antarctica Lipase B (commercially available as Novozyme 435) serves as catalyst for the reaction. To precisely describe the reaction kinetics and the vapour-liquid-equilibrium extensive experimental measurements were conducted at the Institute of Process and Plant Design (Egger, Fieg 2016). The reaction is assumed to only take place in the liquid phase and is modelled by a second order power law.

$$r(T) = k(T) \cdot a_{BuAc} \cdot a_{HeOH} \cdot m_{catalyst} \quad (1)$$

$$k(T) = k_0 \cdot e^{\frac{-E_A}{R}(\frac{1}{T}-\frac{1}{T_0})} \quad (2)$$

UNIQUAC parameters for each binary system were correlated from the results of detailed measurements of the vapour-liquid-equilibrium with a modified ebulliometer.
Furthermore, an accurate calculation of the pressure drop is of high importance. Therefore, correlations were derived from experiments conducted by Ehlers et al. (2017).

$$\frac{\Delta p}{\Delta z} = CP_1 F^{CP_2}(1 + CP_3 w_L) \quad (3)$$

The vapour split is calculated based on the pressure drop in the prefractionator (PF) and main column (MC).

$$VS = 0.5 + constant \cdot (\Delta p^{PF} - \Delta p^{MC}) \quad (4)$$

2.2 Identification of sensor positions

Utilizing the introduced steady state model, suited sensor positions can be chosen for the temperature control. Therefore, sensitivity analyses are conducted, as proposed by Kaymak and Luyben (2005). In this procedure disturbance variables are varied in a small range, typically between 0.1 – 1 %. Steady state simulations are conducted and the sensitivity of the controlled variables is examined afterwards. For the choice of suited sensor positions a high sensitivity is crucial. Furthermore, the distance between the controlled variable and the manipulated variables should be as small as possible, in order to provide a direct and fast response in case of disturbances.
In this work the examined disturbance variables are the heat duty and the feed flow. Each was varied by ±0.5 % and the sensitivity of both temperature profiles, in the prefractionator as well as in the main column, were analysed. In *Figure* 2 a representative sensitivity analysis for the variation of the heat duty is shown.

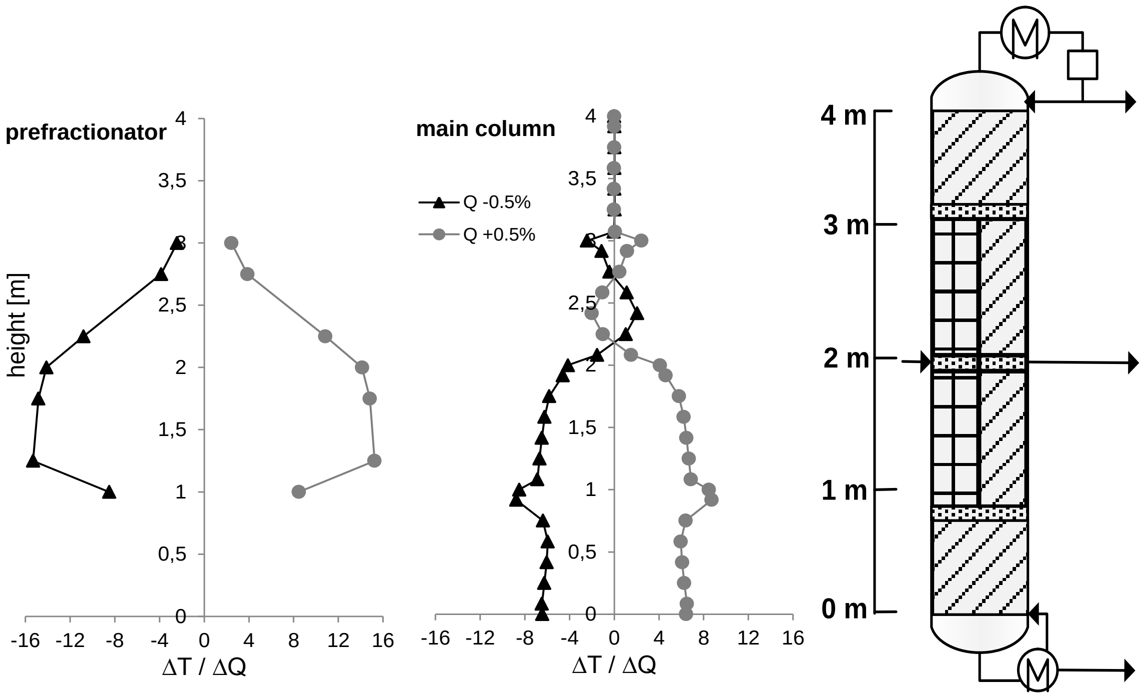

Figure 2: Sensitivity analysis for identification of suited sensor positions for temperature control, temperature sensitivity in the prefractionator and the main column is shown after the heat duty (Q) is varied ±0.5 %

It can be seen that the temperature in the prefractionator is highly influenced by small changes of the heat duty. The stages below the feed stage (below 2 m) show an even higher sensitivity than the stages above. On the other hand these stages are influenced by the feed flow, which is added at 2 m. Thus, the first stage above the feed (height of 2.25 m) is chosen as sensor position for temperature control in the prefractionator. The temperatures in the main column are mainly sensitive in the lower part of the column. The sensitivity above the liquid split (height of 3 m) is very low. Due to the proximity to the manipulated variable, the side stream, a temperature in the separation section below the side stream (below 2 m) is selected as controlled variable. The sensitivity analysis was also conducted for a variation of the feed flow. The resulting sensor positions match the results of the heat duty study. The analysis is performed for each operation point individually in order to find the most sensitive sensor positions for each configuration.

3. Experimental studies

The Institute of Process and Plant Design at the Hamburg University of Technology has built an enzymatic catalysed RDWC in pilot scale. This pilot plant allows investigations of the real operation performance of a RDWC. In this work the pilot plant was operated in closed loop configuration.
As the reaction is catalysed by immobilized Candida antarctica lipase B, enzyme deactivation at elevated temperatures has to be taken into account. Therefore, the reaction and separation process is carried out under vacuum conditions. The operating pressure is 23 mbar.

3.1 Pilot plant

A scheme and a photo of the real pilot plant are shown in *Figure 3*. The column is built in a Petlyuk configuration. The whole plant has a height of 12 m. The diameter in the dividing wall section is DN 50 and in the upper and lower part of the column DN65. The sections S2 and S3 show the reactive section in the prefractionator (with 2 m of reactive packings, type Katapak-SP11). Sections 1, 4, 5 and 6 belong to the main column (with 4 m structured packings, type Montz-Pak-B1-500). Each section is separated by a collector/distributor in order to ensure an even distribution of liquid on all stages. The reflux is divided onto the prefractionator and the main column by the liquid split. For each experiment the required liquid split can be adjusted manually. A vacuum pump at the top of the column enables the operation under vacuum conditions.

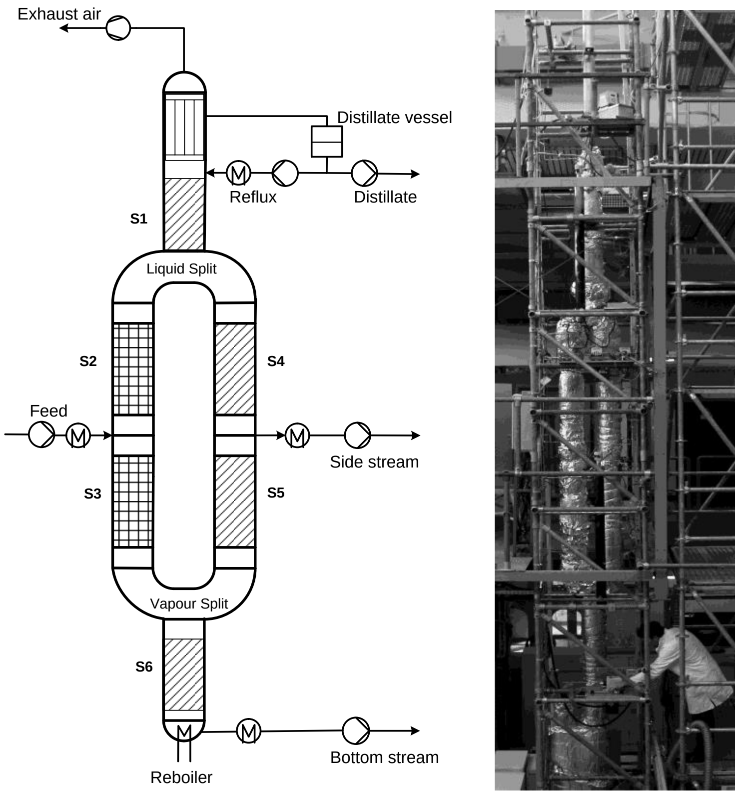

Figure 3: RDWC pilot plant at the Institute of Process and Plant Design at the Hamburg University of Technology

3.2 Equipment for measurement and control

The pilot plant is equipped with a high number of sensors. The large amount of data allows a comprehensive analysis of the processes in the column. In each packing section the temperature profile is measured by four temperature sensors. In total 32 temperature sensors are installed. Furthermore, the pressure drop between the top and the feed stage as well as the pressure drop between top and bottom are measured. The absolute pressure is measured at the top of the column. Levels are determined by two sensors based on the reflexion of high frequency microwaves. Finally, all mass flows in the column are measured by a total of five mass flow meters. To examine the course of the product composition, samples are drawn from all three product flows in defined intervals. The composition of the samples is then analysed by gas chromatography.

A customized process control system is used to monitor and control the operation of the pilot plant. For the conduction of the experiments the control structure was set according to the structure described in Chapter 2.

3.3 Experiments

In order to systematically analyse the dynamic performance of the controlled RDWC several experiments with different disturbance scenarios were conducted. As columns are usually part of a whole production process, they are exposed to fluctuations in the feed flow or the utility. Therefore, five temporary disturbances of the heat duty and feed flow were examined. Furthermore, a permanent disturbance of the heat duty was studied to examine the capacity of the control structure. Moreover, the dynamic performance during set point changes was investigated by setting new values for the controlled temperatures.

Since discussing all conducted experiments would go beyond the scope of this article two selected results will be presented in the following. Therefore, the results of a temporary disturbance in the heat duty and a set point change are selected. *Figure 4* shows the course of the controlled variable and the manipulated variable of the control loop in the prefractionator with two temporary disturbances in the heat duty. It can be seen that the temperature can be stabilized after each disturbance and the process returns to steady state after about 1 hour. The product composition stays constant in the specification. In *Figure 5* the course of the controlled temperature in the main column and the corresponding manipulated variable, the side stream, are shown. In this experiment a new set point of 62 °C was defined. *Figure 5* illustrates that the set point change is adjusted in only a few minutes and with high accuracy. The new temperature is maintained at the desired set point, while the side stream adjusts at a new corresponding value.

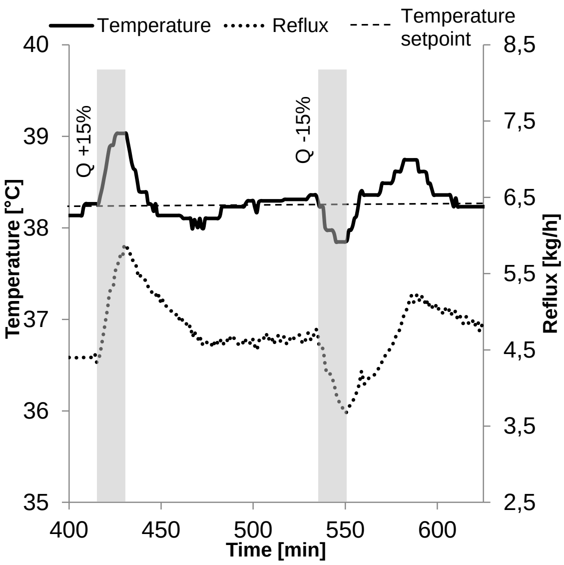

Figure 4: Time course of reflux and temperature in the prefractionator with temporary disturbances of the heat duty (Q) by ±15 %

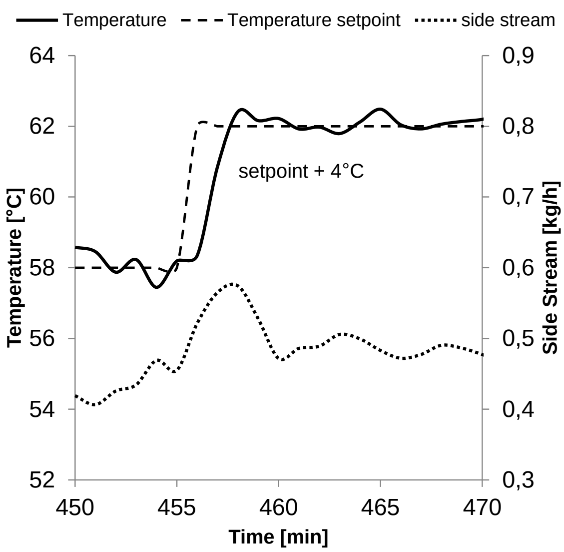

Figure 5: Time course of side stream and temperature in the main column with a set point change of the temperature of + 4°C

In further experiments changes in the feed flow were examined. It was found that temporary disturbances in the feed flow can be adjusted by the proposed control structure in 0.5 to 1.5 hours while the desired product compositions are maintained.

4. Conclusion

In this work a decentralized control strategy for an enzymatic catalysed reactive dividing wall column was proposed, systematically configured by sensitivity analyses and experimentally tested in a pilot plant. In order to identify the most suitable sensor positions for the temperature sensors sensitivity analyses were conducted using a detailed and validated steady state model. For the first time experiments regarding the controlled performance were conducted in such extend in a RDWC pilot plant. In this paper the results of experimental studies of temporary changes in the heat duty as well as a set point change are discussed.

The proposed control concept can handle temporary disturbances in the heat duty successfully while product purities are maintained in specification. Also set point changes can be adjusted by the temperature control in short time and with high accuracy. Further experiments showed that disturbances in the feed flowrate can be adjusted successfully, too. This experimental study generates valuable insights about the dynamic performance of a controlled RDWC pilot plant. It is shown that the highly integrated apparatus RDWC can be operated stable and safely even under the influence of disturbances. This is an important step towards the successful adaption of the RDWC in chemical industry.

In a next step we will extend the control concept by including the heat duty and the liquid split into our control concept. Based on our knowledge gained from this work we will design control concepts which enable the energy optimal operation of RDWCs. The advanced concepts will be analysed in dynamic simulations and finally be tested on our pilot plant.

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