Experimental Characterization of an Innovative Separation Tray with Enhanced Flexibility

Julia Riese [a], Henrik Fasel [a], Marcus Grünewald [a]

[a] Ruhr University Bochum, Laboratory of Fluid Separations, Universitätsstr. 150, 44801 Bochum, Germany
riese@fluidvt.rub.de; fasel@fluidvt.rub.de; gruenewald@fluidvt.rub.de

Abstract. In the past, the need for flexible equipment has increased in chemical industry. However, unit operations like distillation are characterized by distinctive limitations restricting flexible operation. Thus, there is a need for innovative equipment that allows for an enhanced flexibility. Therefore, we developed and investigated a tray column that was designed for flexible operation. The column consists of various segments within one column shell that can be operated independently. Here, we presented a hydrodynamic characterization of a single segment operated at different steady-state operating points. Furthermore, the single segment was operated under liquid transfer to an adjacent segment. The latter operation mode was developed for a suitable start-up strategy of single segments. Results in terms of pressure drops indicated a stable operation of the single segment at various operating points. Nevertheless, the operation under liquid transfer superimposed additional operation limits that are investigated in more detail in the future.

Keywords Tray Design, Cold Flow Experiments, Fluid Dynamics, Flexible Operation

Introduction

Ensuring future competitiveness of production processes is an important task for the chemical industry. This requires the processes to be constantly and adequately adapted for the related challenges. These challenges result, among others, from the need to reduce greenhouse gases significantly. In particular, future energy and feedstock supply will increasingly rely on renewable rather than on fossil resources. Especially, an energy supply structure that heavily relies on intermittent renewable resources imposes new challenges for the operation of chemical production processes.

Adjusting the operation of the processes to the availability of energy streams is possible by enhancing the flexibility of processes and their unit operations [1]. In literature, various solution strategies to enhance process flexibility can be found, e.g. for chemical reactors [2-4]. This also applies to downstream processing unit operations such as distillation columns [5]. In fact, distillation processes consume a considerable amount of energy, so that energy- and separation-optimal operation needs to be ensured at all operating points. Technical solutions are required to cope with these increasing uncertainties.

In our work, we have developed a novel column design that allows for a more flexible operation of distillation processes [6]. The column and the associated tray layout is illustrated in Fig. 1. The internal volume of the column is segmented by installing vertical dividing walls throughout the total height of the column. Every segment in the column can be operated, started-up and shut-down independently. In fact, each segment can be operated at a distinctive separation-optimal operating point, which is the basis for the increased flexibility.

The segmentation of the column resulted in two specific tray layouts to achieve efficient contact between gas and liquid on each tray. As shown in Fig. 1, the tray layouts differ in the flow direction of the liquid over the active area of the tray and therefore in the positioning of the weir and the downcomer. In the tray layout D_I, the liquid flows from an inner laying downcomer in radial direction over the active area to an outer weir. On the tray with the layout D_O, the flow direction of the liquid is *vice versa* and the liquid is entering the active area from an outer laying downcomer towards an inner weir.

Besides these distinctive tray layouts, the column is characterized by the capability of coupling the segments on each tray via the downcomer next to each other. The motivation behind this coupling is to accelerate start-up operation of single segments. If one segment is operated at steady-state, the start-up of the adjacent segment can be supported by liquid transferred from the downcomer of each tray. Thus, an inactive segment is started-up using a pre-defined concentration profile. Bruns *et al.* [7] developed a dynamic column model to perform start-up simulations, analyzing this approach to start-up individual segments within one column shell. Results show that a significant reduction of the start-up time is possible, while the active segment is affected only for a short time. During start-up, only <10% of the liquid holdup from the active downcomer was used. Anyway, to validate these simulation results, it is necessary, to

assess the liquid transfer from one active downcomer to the adjacent inactive ones in experimental studies. In addition, insights into the specific operation of single segments in this column design are yet to be discovered.

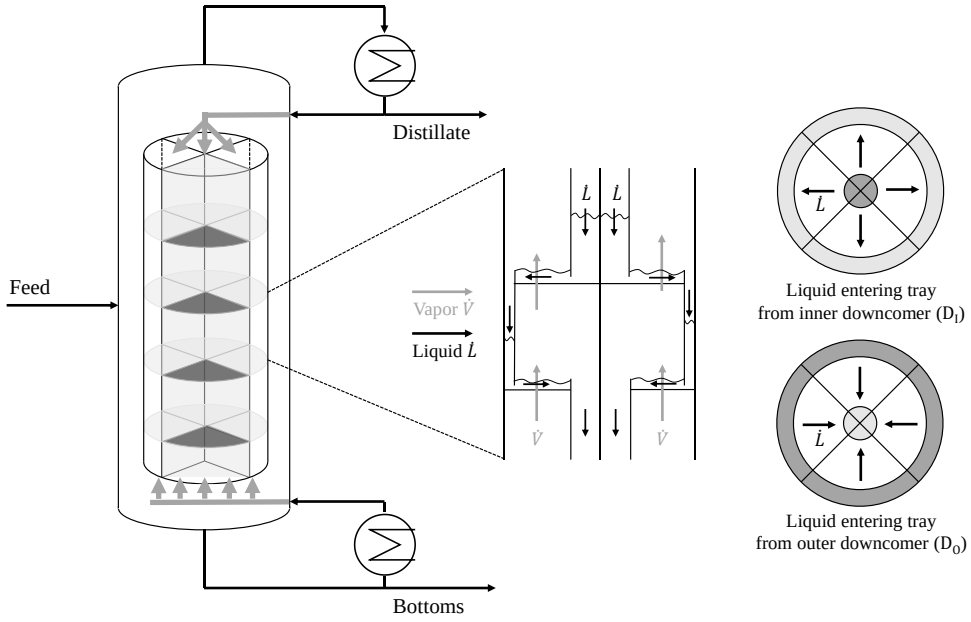

Fig. 1 Schematic representation of the flexible column (left), flow directions in the segments (middle) and tray layouts (right).

Therefore, the aim of this contribution is to perform a hydrodynamic characterization for one single segment in the flexible column, being operated at varying gas and liquid loads. Additionally, we quantify the amount of liquid that can be transferred between an active segment and an inactive adjacent one without compromising stable operations. Finally, we assess the hydrodynamics on the trays and in the downcomer during operation with liquid transfer.
We start this article by presenting the configuration of the column and trays, as well as the experimental set-up. We then describe and discuss the results of our experimental investigations, followed by a summary of our findings and an outlook on future research activities.

Materials and Methods

The experiments are carried out in a PVC tray column with an inner diameter of $d_c = 0.6$ m. As shown in Fig. 1, the cross-sectional area of the column is divided into four segments with equal active area and the column is equipped with sieve trays. A more detailed description of the experimental set-up is addressed in [8].
The column is constructed in such a way that different trays with different hole diameters and opening ratios, respectively, can be installed. For the experiments that are presented here, the trays have a hole diameter of $d_h = 10$ mm. Taking the active area of a single segment and the perforated area ($A_h = 3.68 \cdot 10^{-3}\ m^2$) into account, this results in an opening ratio of $\varphi = 5.7\%$ for both tray layouts. All relevant geometric data for the column and the different tray layouts, D_O and D_I, is summarized in Table 1.

Table 1 Design parameters of the column and the different tray layouts.

Parameter	Tray D_O	Tray D_I
Tray spacing S /m	0.5	
Column diameter d_c /m	0.6	
Tray thickness s_t /m	0.005	
Active area per segment A_a /m²	0.0565	
Cross section area per segment A_S /m²	0.071	

Weir height h_w /m	0.046	0.030
Weir length l_w /m	0.152	0.442
Height downcomer clearance h_c /m	0.002	0.006

The experiments were carried out using the test system water / air at atmospheric pressure and ambient temperature. Fig. 2 depicts the flowsheet of the experimental setup that was used to characterize the hydrodynamics of one single segment with and without liquid transfer.
Gas was supplied by an electrical fan and adjusted by rotational speed, resulting in gas flow rates between 75 $m^3 \cdot h^{-1}$ and 420 $m^3 \cdot h^{-1}$. The gas load factor F_v is calculated according to equation (1), where $u_{g,h}$ represents the gas velocity in the holes of the sieve trays and ρ_g the gas density.

$$F_v = u_{g,h} \cdot \sqrt{\rho_g} \cdot \frac{A_h}{A_S} \quad (1)$$

Thus, gas load factors, or F-factors, range between 0.3 $Pa^{0.5}$ and 1.8 $Pa^{0.5}$. Liquid flow rates were varied between 1.6 $m^3 \cdot h^{-1}$ and 5.5 $m^3 \cdot h^{-1}$. Usually, for sieve trays, the liquid load is defined per meter weir length. For the particular tray design used in the flexible column, however, liquid load is specified per m^2 active area to ensure comparability between the tray layouts. This resulted in liquid loads between 25 $m^3 \cdot m^{-2} \cdot h^{-1}$ and 105 $m^3 \cdot m^{-2} \cdot h^{-1}$. Gas flow rates were determined using a metering orifice, for which pressure drops were measured and evaluated according to a standard procedure. Liquid flow rates were set using standard rotameter.

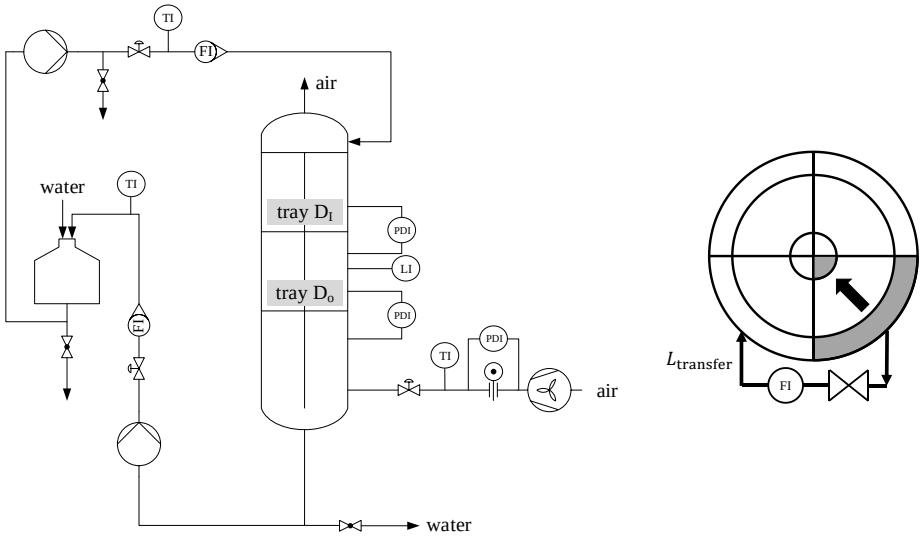

Fig. 2 Flowsheet of the experimental set-up with one segment operated and illustration of liquid transfer between the segments.

The aim of the experiments was the measurement of dry pressure drop and overall pressure drop of the two-phase flow at different gas and liquid loads as well as different liquid transfer rates between the segments. Therefore, the experimental set-up was equipped with standard u-pipe manometers for higher expected pressure drops and additionally with inclined u-pipe manometers to enhance reliability of the measurements at lower expected pressure drops. In addition, we measured the clear liquid height in the downcomer of the tray layout D_O during liquid transfer from the downcomer.
Liquid transfer was facilitated using downcomer trapouts according to Kister [9]. Three trapout pipings were installed at the outer shell of tray D_O and thus from the outer downcomer of the tray D_O and before entering the active area of the tray. The total cross sectional area of the three trapouts sums up to $A_t = 24 \cdot 10^{-4}$ m^2. The liquid transfer is illustrated in Fig. 2. We measured the liquid flow rates transferred from the downcomer of the tray D_O in the active segment with an attached turbine wheel flow meter. The amount of liquid that can be transferred depends on the liquid load in the active segment, the liquid load in the adjacent one, the cross sectional area and the valve opening. During

the experiments with liquid transfer, liquid height and thus pressure drop was additionally monitored in the downcomer of the tray D_O. Parallel monitoring of the liquid height in the inner laying downcomer of tray D_I was not possible due to complex configuration of the overall column layout.
All measurements were repeated at least three times. The results presented in the next section represent mean values for all measurements at each single operating point. Relative standard deviations were within a range of <10% for almost all measurements. Ensuring readability of the resulting plots, relative standard deviations are only shown if necessary for the discussion.

Results

First, we present the results of the hydrodynamic characterization for the operation of one single segment. Therefore, pressure drops, including the dry pressure drop for a liquid flow rate of $L = 0\ \mathrm{m^3 \cdot m^{-2} \cdot h^{-1}}$, are shown in Fig. 3 for both tray layouts, D_I and D_O. It can be seen that, as expected, dry pressure drops (Δp_{dry}) are significantly lower than the pressure drops for the two-phase flow. In addition, the dry pressure drops do not significantly differ between the tray layouts D_I and D_O. For the dry pressure drop, we find a good agreement with the correlation of Bennet [10].

$$\Delta p_{\mathrm{dry}} = \frac{a}{c_v^2} \cdot u_{\mathrm{g,h}}^2 \cdot \rho_{\mathrm{g}} \qquad (2)$$

The pressure drop coefficient was determined using geometric data of the tray layouts and resulted in $c_v = 0.82$. A value of $a = 0.499$ for the correlation constant was chosen according to the approximation by Leibson *et al.* [11]. Errors between the correlation and the experimental results are <10% for both tray layouts.
Looking at the pressure drops at different liquid loads, significant differences can be observed for both tray layouts. On tray D_O there is a strong dependency between pressure drops and liquid loads. With increasing liquid loads, increasing pressure drops are measured over the total range of gas load factors. On the tray D_I, pressure drops are almost independent from the liquid load over a wide range of gas load factors. One possible explanation can be attributed to the different weir heights for both tray layouts (cf. Table 1). The weir of the tray D_I is 30% lower than on the tray D_O. Thus, accumulation of liquid in front of the weir is less pronounced. In addition, due to the increasing radius in flow direction, less turbulence can be observed in the two-phase flow for all liquid loads investigated.

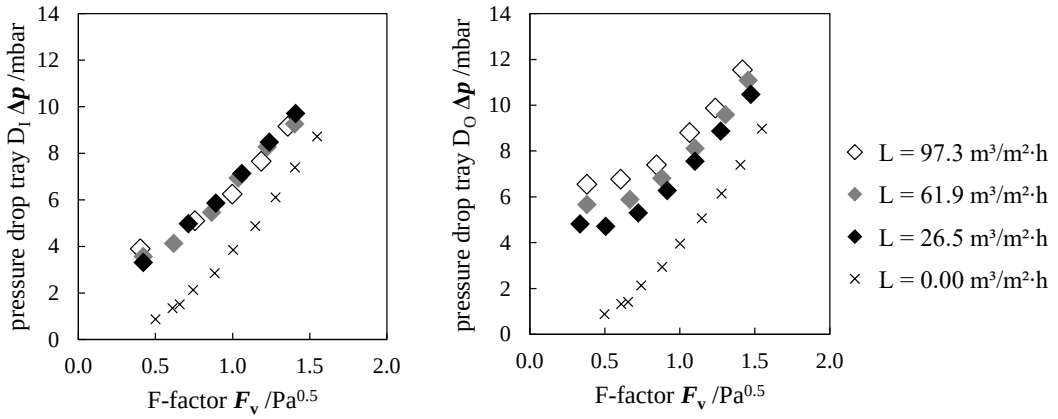

Fig. 3 Dry and overall pressure drop for the tray design D_I (left) and D_O (right) as a function of gas load factor and for different liquid loads.

In a next step, we identified the liquid flow rates that can be withdrawn from the active segment during operation, as this is an important factor for the coupling of the segments during operation. The main driving force for the liquid transfer is the pressure difference between the active and the adjacent inactive segment. If the inactive segment is at atmospheric conditions without any liquid, this driving force is at its peak. Therefore, to identify the maximum possible transfer rates, we did not actively operate the adjacent segment to which the liquid is transferred. The liquid was drained from the outer downcomer of tray D_O before passing through the downcomer clearance and entering the active area of the tray. Thus, the share of liquid that was transferred was not passing over the active area, leading to a decrease of the specific liquid load on this tray.

Fig. 4 shows the share of liquid that can be withdrawn from the downcomer of tray D_O for a fixed cross-sectional area of the trapouts and different valve openings at a liquid load of $L = 97.3\ m^3 \cdot m^{-2} \cdot h^{-1}$.
The amount of liquid that can be withdrawn is, as expected, a function of the valve opening. With this experimental set-up, a maximum liquid transfer of 17.5% with respect to the total liquid flow rate ($L = 5.5\ m^3 \cdot h^{-1}$) is possible. A further increase of the liquid draw-off is possible by enhancing the cross-sectional area of the trapouts. At lower liquid flow rates, lower transfer rates were achieved. Anyways, for a valve opening of 100%, a minimum of 10% of the liquid flow rate can be transferred to the adjacent segment for all investigated liquid loads. This is in accordance with the required liquid transfer rate that resulted from the start-up simulations [7].

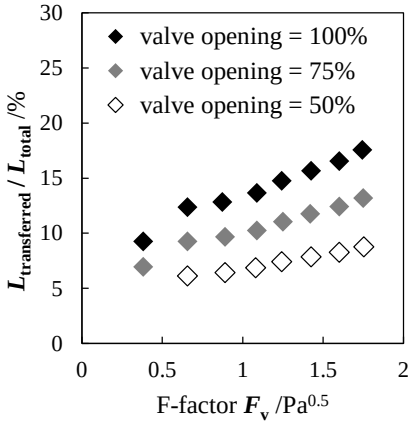

Fig. 4 Amount of liquid transferred as a function of gas load factor and for different liquid loads in the segment under operation.

Finally, we assessed the pressure drops during operation with liquid transfer to identify the influence of the withdrawal of liquid from the downcomer of tray D_O on the operation of the trays and the downcomer. Fig. 5 shows the overall pressure drops in the downcomer of tray D_O as well as on both trays during the operation with liquid transfer at a valve opening of 100%. Results are shown for two liquid loads in the active segments, namely $L = 97.3\ m^3 \cdot m^{-2} \cdot h^{-1}$ and $L = 26.5\ m^3 \cdot m^{-2} \cdot h^{-1}$. Stable operation of the trays is ensured if the following stability criterion is met at all operating points.

$$\Delta p_{\text{downcomer}} > \Delta p_{\text{tray}} \tag{3}$$

The pressure drop in the downcomer has to be higher than the pressure drop on the active area of the trays to ensure a sufficient driving force for the liquid to enter the tray and flow over the outer weir.

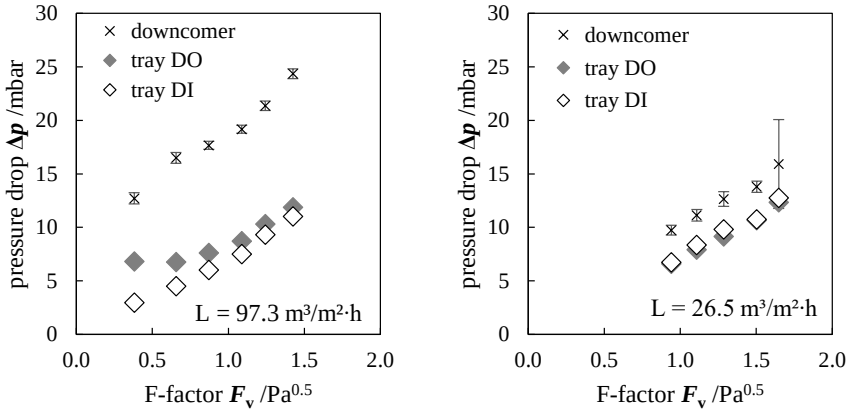

Fig. 5 Pressure drops in the downcomer and on the trays Do and Di during operation with liquid transfer at different liquid loads in the segment.

From Fig. 5 it becomes apparent that the stability criterion is ensured at high liquid loads ($L = 97.3\ m^3 \cdot m^{-2} \cdot h^{-1}$) over the total range of investigated gas load factors. On the contrary, at lower liquid loads ($L = 26.5\ m^3 \cdot m^{-2} \cdot h^{-1}$)

the difference between pressure drop on the trays and in the downcomer decreases, mainly because the liquid level in the downcomer decreases due to the liquid transfer. This leads to operating points, especially at increasing gas load factors within the segment, where the liquid level in the downcomer is highly fluctuating as indicated by the largely increasing error bar for the downcomer pressure drop. At these operating points, the stability criterion cannot be ensured safely due to possible downcomer blowing. In conclusion, the need for a safe and stable operation superimposes additional limitations regarding the amount of liquid that can transferred from one segment to the adjacent one.

Conclusions

In this work, we presented results for an experimental characterization of a flexible tray column design. We investigated the operation of a single segment of this tray column design in terms of pressure drops for various gas and liquid loads. Additionally, we investigated the operation of a single segment while parts of the liquid are transferred to an adjacent inactive segment. This operation mode was developed for the start-up of segments with a pre-defined concentration profile. Therefore, on each tray, liquid was transferred from a downcomer in the active segment to the adjacent downcomer in the inactive segment.

Results showed that a stable operation of a single segment is possible in a broad range of liquid load and for all investigated gas load factors. Furthermore, it was possible to transfer liquid from an active segment during operation to facilitate the aforementioned start-up strategy for coupled segments. Nevertheless, distinctive operation limits resulted from the operation with liquid transfer to prevent downcomer blowing, in particular at low liquid loads and increased gas load factors.

In future work, we want to extend the hydrodynamic characterization for both steady-state operation and operation during liquid transfer. Characteristic parameters such as the liquid height on the both tray layouts and the liquid distribution along the flow path on both tray layouts are assessed. Further operating points need to be investigated in order to achieve a full picture regarding the operating window of the individual segment. Additionally, a thorough characterization of mass transfer performance is planned.

Acknowledgements

The authors acknowledge financial support by the Federal Ministry of Education and Research, Germany, in the project TransProMinC (project number 01LN1712A). The authors also thank the Max Buchner Research Foundation under grant number 3787.

References

1. J. Riese and M. Grünewald, Challenges and Opportunities to Enhance Flexibility in Design and Operation of Chemical Processes. *Chemie Ingenieur Technik*, **92**, 1887-1897 (2020).
2. J. Maußner, C. Dreiser, O. Wachsen and H. Freund, Systematic model-based design of tolerant chemical reactors. *Journal of Advanced Manufacturing and Processing*, 1:e10024 (2019).
3. K.L. Fischer, H. Freund, On the optimal design of load flexible fixed bed reactors: Integration of the dynamics into the design problem. *Chemical Engineering Journal*, **393**, 124722 (2020).
4. B. Bruns, F. Herrmann, M. Grünewald and J. Riese, Dynamic Design Optimization for Flexible Process Equipment. *Industrial & Engineering Chemistry Research*, **60**, 7678-7688 (2021).
5. C. Hoffmann, J. Weigert, E. Esche and J.-U. Repke, Towards demand-side management of the chlor-alkali electrolysis: Dynamic, pressure-driven modeling and model validation of the 1,2-dichlorethane synthesis. *Chemical Engineering Science*, **214**, 115358 (2020).
6. J. Riese and M. Grünewald, Flexibility for Absorption and Distillation Columns. *Chemical Engineering Transactions*, **69**, 787-792 (2018).
7. B. Bruns, H. Fasel, M. Grünewald and J. Riese, Development of a Dynamic Modeling Approach to Simulate a Segmented Distillation Column for Flexible Operation. *ChemEngineering*, **5** (2021).
8. H. Fasel, M. Grünewald and J. Riese, New Column Design to Enhance Flexibility: Concept for Hydrodynamic Characterization. *Chemie Ingenieur Technik*, **92**, 2035-2040 (2020).
9. H.Z. Kister, *Distillation Operation*. McGraw Hill Education, McGraw Hill, New York (2013).
10. D.L. Bennett, R. Agrawal and P.J. Cook, New pressure drop correlation for sieve tray distillation columns. *AIChE Journal*, **29**, 434-442 (1983).
11. I. Leibson, R.E. Kelley and L.A. Bullington, How to Design Perforated Trays. *Petroleum Refiner*, 36, 127-133 (1957).