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Flexibility for Absorption and Distillation Columns

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The flexibility of absorption and distillation columns is limited by macroscopic fluid dynamic phenomena like flooding, entrainment, weeping or dewetting. The design of those columns is a crucial task and depends on the separation task, the needed capacity and the fluid dynamic boundaries. An enhancement in flexibility is necessary to face upcoming challenges in chemical industry, including increasing uncertainties in material and energy supply or product demand. Results show that an increased flexibility is either possible by means of internal structuring or modularization of the columns. Both measures have a potential to enhance capacity flexibility of absorption and distillation columns.

1. Introduction

The chemical process industry is facing various challenges, which require higher adjustability of processes to imposed changing boundary conditions. Those challenges are increasing product differentiation and increasing uncertainties in market forecasts (Buchholz, 2010). Additionally, rising shares in the use of volatile renewable energy cause increasing uncertainties regarding availability and price (Riese, 2016). Those challenges lead to higher requirements regarding the flexibility of chemical processes. Commonly used definitions of flexibility in process engineering are product flexibility, feedstock flexibility and capacity or volume flexibility (Kranenburg et al., 2015; Seifert et al., 2014; Rauch, 2003), whereas the latter is in focus. Taking into account the overall production plant, the capacity flexibility of the process equipment is limiting the flexibility of the overall plant (Seifert et al., 2014). Therefore, the limiting factors and possibilities to overcome restrictions in capacity flexibility are discussed for two scenarios: distillation column for large-scale continuous plants and absorption column for small-scale modular plants. Modular plants demonstrated their advantages for processes especially in the production of pharmaceuticals and fine chemicals (Buchholz, 2010) and their economic benefits are shown in different studies (e.g. Lier et al., 2011). An example for the application of large scale distillation columns having a high capacity flexibility are Power-to Fuels processes in which capacity is a function of available renewable electricity to produce hydrogen via water electrolysis.

2. Fluid Dynamics

To discuss the implications of varying vapour and liquid flow provoked by varying feed flows, it is important to be aware of the fluid dynamics of conventional columns used in chemical industry. Most important are tray or packed columns for distillation and absorption processes. While tray columns are characterized by low costs and well described rules for design and scale-up, packed columns do have considerably lower pressure drops (Lockett, 1986). Additionally packed columns are characterised by fewer degrees of freedom in the design procedure (Kiss, 2013).

To ensure safe operation it is necessary to describe the operation range for the separation performed within the column. The operation range is characterized by different limits regarding the macroscopic fluid dynamics within the column. Associated performance diagrams are used to visualize the operation range and deduce the possible capacity flexibility. Figure 1 shows the operation range for a tray distillation column. Within the performance diagram, the vapour ($\dot{V}$) and liquid ($\dot{L}$) flows are normalized by the active area (A_a) of the column.

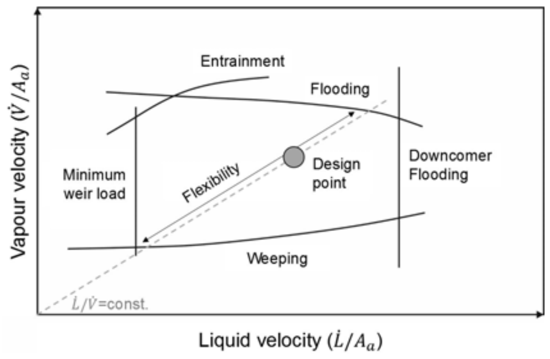

Figure 1: Performance diagram and operation limits for tray columns (adopted from Düssel & Stichlmair, 2006).

As illustrated by the performance diagram, four main phenomena limit the operating range of a tray column. The maximum vapour and liquid velocities are indicated by flooding of the column. Flooding means that the liquid is accumulating in the column and can be observed by a strong increase of pressure drop. Two types of flooding are possible: downcomer flooding and jet flooding. Backing up of liquid in the downcomer and thus pushing back over the weir of the tray above causes downcomer flooding. Downcomer flooding occurs at high vapour and liquid velocities and is supported by too small tray spacing. In contrast to this, jet flooding is caused by excessive entrainment of the liquid due to high vapour velocities. The actual mechanism of entrainment depends on the flow regime of the two-phase flow on the tray (Lockett, 1986). Weeping is associated with low vapour velocities which results in liquid descending through the tray perforations towards the subjacent tray. Low liquid velocities lead to situations in which there is not enough liquid passing over the tray leading to maldistribution of the liquid and decreasing efficiencies. In practice, it is usual to ensure a height of the liquid over the weir of 0.05 m (Stichlmair & Mersmann, 1978). The design point within the operation range can be influenced by the design and operation variables of the column and depend on the separation task as well as the needed capacity, which is limited by fluid dynamics. Additionally, there is a trade-off between efficiency and capacity (Kiss, 2013).

Mass transfer in tray columns mainly occurs in the froth on the tray while liquid is flowing over the tray and vapour is rising through the tray orifices. Phase boundary area for the mass transfer is available due to small vapour bubbles dispersed in the liquid flowing over the tray. Thus, bubble or froth regimes are the preferred flow regimes for the operation of tray columns (Lockett, 1986; Kister, 1992). In contrast, in packed columns mass transfer takes place between the upward gas flow and a downward liquid film that is distributed over the random packings and structured packings, respectively. Using random packings, liquid droplets also play a crucial role for mass transfer. Thus, leading to specific fluid dynamic limitations. Dominating fluid dynamic parameters for packed columns are pressure drop and relative liquid hold-up. Depending on the operation point, both parameters are a function of the liquid load and the F-Factor (F_v). For common processes packed columns are operated between the loading line and flooding point, typically at 65 – 80 % of $F_{v,flooding}$ (Maćkowiak, 2003). Here, pressure drop and liquid hold-up increases with increasing liquid load and increasing F-Factor. At high ratios between gas and liquid loads steady state counter-current flow is no longer possible due to high interactions between gas and liquid causing flooding of the column. Flooding is also possible due to excessive entrainment of the liquid. This occurs at lower F_v values, compared to the aforementioned flooding mechanism, and high liquid loads and limits the operation of the column. For packed columns, flooding is also indicated by a strong increase in pressure drop. In contrast to that, the minimal load of a packed column is indicated by dewetting of the packings. In this case, the liquid flow is too low and the overall surface area of the packings is not used. (Billet, 1995). In packed columns, the measures for gas and liquid distribution are more important because there is an inherent relationship between gas and liquid loads, surface area and mass transfer efficiency.

3. Enhancement of Flexibility by Tray and Column Design for Conventional Distillation Columns

From scientific literature, no concepts to enhance capacity flexibility of conventional columns are available. Anyhow, by conducting a review on patents various concepts are found. The patents that explicitly cover varying vapour and liquid loads within the column are categorized into two different measures to ensure safe operation. One group of patents addresses the design of the trays to enhance the range of vapour and liquid flow that can be handled during operation. The other group deals with measures regarding an internal segmentation of the column to reach the same goal. Some of those patents are described in Table 1.

Table 1: Examples of patents dealing with enhancement of operation range in distillation columns.

Patent no. (year)	Constructive measure	Effect on fluid dynamics
US 2,692,128 (1954)	Arrangement of bubble caps in different heights on each tray	Robust two-phase flow regime by operation with very low vapour and liquid loads
US 4,578,153 (1984)	Dividing the tray into sectors with various weirs, leading to different clear liquid height	Realising very low vapour loads by means of different drags within each sector
EP 01 29 198 B1 (1989)	Bubble cap having two zones of slots on different levels	Safe operation if vapour and liquid loads vary in large ranges
DE 44 44 891 C1 (1997)	Segmentation of batch distillation column into hermetically sealed segments	Allowing for an operation in a wide range of batch feed

The following concept, which was analysed in detail, combines both presented measures to enhance the capacity flexibility of a conventional distillation column. By an internal structuring of the vapour and liquid flow, the column was divided in individual segments that can be operated independently. To ensure equal liquid flow above the tray the liquid was assumed to flow in a radial direction, alternating from the centre of the column towards the edge and from the edge toward the centre. The concept is illustrated in Figure 2 (left). The possible enhancement of flexibility was evaluated by simulation studies using AspenPlus®. The implemented simulation structure is shown in Figure 2 (right). Comparability was ensured by taking the active area of the column as scaling parameter into account. For the evaluation it was assumed that each segment had a separate condenser and reboiler. Analysis of concentration profiles within each segment shows that an individual feed to each segment leads to more uniform separation efficiency compared to using only one feed.

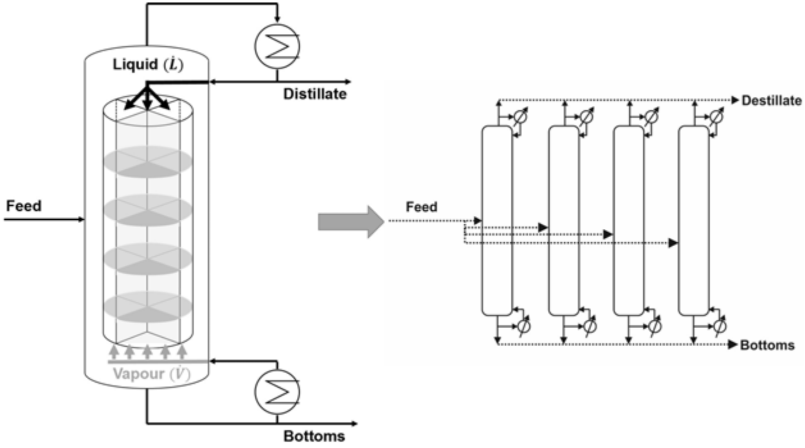

Figure 2: Schematic illustration of concept to enhance flexibility of distillation columns (left) and the simulation structure for four segments (right).

The structured column was investigated for different numbers of segments but a fixed separation task. The enhancement in capacity flexibility was illustrated by reference to a base case, whereas the base case was a conventional column needed for the separation task (see Table 2).

Table 2: Separation task and corresponding operating parameters used for the evaluation of presented concept.

Separation task		Operating parameters	
Binary mixture	Chlorbenzene / Ethylbenzene	Number of theoretical stages	30
Composition	equimolar	Feed stage	15
Feed flow	10,000 kg/h	Reflux ratio	11.4
Caloric value	e=1	Pressure drop	0.21 bar
Pressure	atmospheric	Active area	5.65 m²

The simulation-based analysis of this concept shows that it was possible to enhance the variation in capacity significantly by structuring the column. The study was performed by varying the feed flow to the column systematically. Simultaneously, all operation parameter were constant and the product specifications defined by the base case were achieved. The operation range was specified for each scenario. Thus, the relative limits for each segment were the same. Anyway, the absolute limits were a function of the number of segments within the column. The design parameters and areas in the columns were adjusted in a way that the active area was always comparable to the conventional column. The design point of the conventional column allowed a variation of capacity of 51 % around the design point. By enhancing the number of segments within the column this variation increased. For example by using four segments a variation of roughly 120 % was feasible. (Riese 2016) The results are summarized in Figure 3.

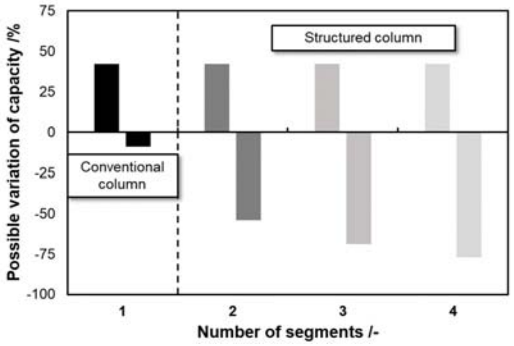

Figure 3: Comparison of capacity variation within the operation ranges of a conventional column and a structured column as a function of the number of segments.

With these simulations it was also possible to identify switching points for the commissioning of new segments depending on the feed flow. For the future an experimental investigation of the concept is planned. Therefore, aspects like the liquid distribution in each segments and on the trays, a vapour split mechanism and measures for the feed inflow to each segment are treated. The latter is possible for example by using a loop line with valves around the column.

4. Enhancement of Flexibility for Absorption Column by Modularization

For the production of fine and specialty chemicals modular processes are in focus of research and industry for almost 10 years now (Buchholz, 2010). Modular processes are based on modular process equipment allowing for a fast adjustment on changing market situations by numbering-up and equaling-up (Lier et al., 2018). A bottleneck for the industrial implementation so far are modular apparatuses for unit operations like absorption and distillation. In the past, research activities to develop modular process equipment for unit operations focused on concepts involving micro engineering. (Kenig et al., 2013) Anyway, those concepts have a major disadvantage: Due to the micro-scale, many parallel channels are necessary to facilitate sufficient throughput. If mass and heat transfer takes places at free phase boundaries, like in absorption and distillation columns, an equal distribution of gas and liquid flow within each parallel micro channel is a major challenge. Additionally, a very high number of parallel channels lead to high values of fixed capital investment for construction of those.

To overcome those disadvantages, it is necessary to develop concepts that allow reasonable throughput in one singular module. Therefore, Müller et al. developed and investigated a singular absorption module, which is based on a rectangular cross section (Müller et al., 2015). The development is based on the idea to adopt the design of plate heat exchangers for modular separation units. Thus, being able to easily add or replace modules with standardized interfaces in one single rack leading to low space requirements and high mobility. Additionally, using this concept a high capacity flexibility is feasible by numbering-up of the characterized single module. The single module investigated, showed hydrodynamic behaviour and mass transfer efficiencies comparable to conventional columns when using structured packings as column internals (Müller et al., 2015). In further studies, a parallel interconnection of modules, representing a numbering-up scenario, was examined experimentally. One module had a width of 500 mm and a depth of 100 mm. The height of the structured packings (Raschig Super Pak 150Y) was 0,95 m. Within the experiments, liquid loads up to $u_l = 40\ m^3/m^2h$ and F-Factors up to $F_v = 4\ Pa^{0,5}$ were realized. In the experimental set-up, each modules had an individual but similar liquid distributer installed above the packings. For the gas flow one inlet and one outlet nozzle was available. Thus, the gas flow through the modules followed a z-shaped course. Perforated plates were installed in each module to allow for sufficient gas distribution. During the experiments, sealing plates were inserted between the modules at the bottom to prevent the gas on entering more than one or two modules, respectively. The experimental set-up is shown in Figure 4.

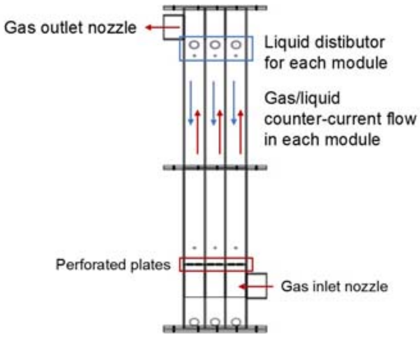

Figure 4: Experimental set-up for three modules operated parallel (adopted from Paul et al., 2016).

Pressure drop, liquid hold-up and mass transfer were examined within the experiments for one, two and three modules operated parallel. Mass transfer experiments were conducted by NH_3-absorption in water. The aim of those experiments was to prove that a capacity expansion is possible by enhancing the number of modules operated. Figure 5 gives an example for the averaged specific pressure drop as a function of the F-Factor in one, two and three modules for a liquid load of $u_l = 20\,m^3/m^2h$. As shown, especially for lower F-Factors the pressure drop was independent of the number of modules being operated parallel. For higher F-Factors the pressure drop differed slightly.

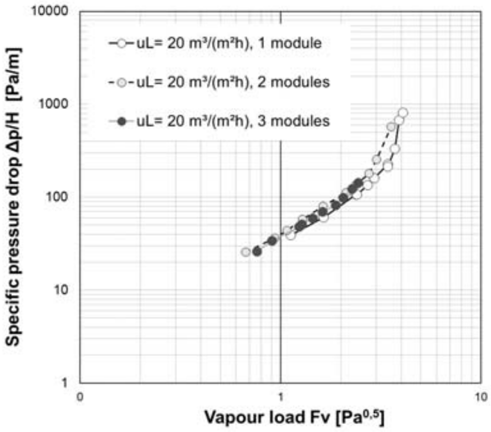

Figure 5: Specific pressure drop as function of F-Factor for varying number of modules and a liquid load of $u_l = 20\,m^3/m^2h$ *(Paul et al. 2016).*

From this results, which were similar for higher and lower liquid loads, it is concluded that there was a proportional increase in gas throughput as a function of the number of modules. For two modules the gas flow was doubled and for three modules a threefold increase in gas flow was possible without changes in the average specific pressure drop. Similar results were obtained for liquid hold-up and mass transfer. Anyway, results for the liquid hold-up show that the loading regime was shifted to higher liquid loads if the number of modules increase. With given experimental set-up it was only possible to reach flooding in case of one module operated. For two and three modules it was not possible to reach flooding. When operating two or more modules parallel, the specific liquid hold-up showed consistent results within each module and for all investigated liquid loads. Nevertheless, there were some drawbacks in mass transfer efficiency if it comes to higher numbers of modules and higher liquid loads. This was due to visibly observable maldistribution of gas and liquid within the modules, which was not tracked by the integral values investigated. Maldistribution is supported by the rectangular design of the column. Therefore, additional measures are necessary to improve phase distribution for numbering-up of the single module.

In conclusion, it was shown that modularization is a possible way to enhance the capacity flexibility of absorption and distillation columns. For the latter unit operation, a transfer of the concept needs to be done and evaluated experimentally.

5. Conclusion

Current and future challenges for the chemical industry impose increasing needs for flexibility in plant operation. A major bottleneck for the flexibility of the production plant is the flexibility of the process equipment itself. Anyway, different measures to increase capacity flexibility for applications on different scales are discussed and show promising results. Segmentation of vapour and liquid flow within a conventional tray column shows significant increase in capacity flexibility. The variation of feed flow is more than doubled by this measure ensuring safe operation of the column at the same time. For small-scale plants, modularization is a suitable tool to enhance the flexibility. A modular design and experimental investigation of an absorption column leads to a concept where capacity is scalable respect with to the number of modules operated parallel by numbering-up of single thoroughly characterized modules. Both presented solutions to enhance the capacity flexibility of unit operations show that an internal structuring of the columns is a promising method to tackle future challenges in chemical production.

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