

Experimental studies on the fluid dynamics of a miniaturized measuring cell for an improved design procedure of distillation columns – concept of the measuring cell

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Abstract: Common design methods of distillation columns necessitate safety margins and thus oversized equipment. Therefore, a more accurate design method is needed and has to be developed. Miniaturizing experimental setups is one possible approach. The concept of miniaturizing based on the research projects at Ruhr University Bochum (RUB) were presented here. After specifying the main aspects, a detailed design of the miniaturized measuring cell for investigating distilled phenomena was described. The measuring cell featured a rectangular 106 x 20 mm cross section and contained two sheets of standard structured packing. This experimental setup was developed within the research project ReProvAP (Reduction of climate-relevant process emissions by an improved design of columns with structured packings).

Keywords measuring cell, miniaturization, distillation column, structured packing, scale-up

1. Introduction

Distillation columns with structured packings are widely used for separation processes, but due to uncertainties in the predictability of the separation performance, the design can lead to oversized equipment. A common strategy to design industrial plants is to investigate test mixtures in lab-scale plants and to employ simulations with simplified assumptions. Based on the determined HETP value a scale-up is then calculated. (1, 2, 3) Among other factors this method is error-prone in the case of non-ideal material systems. To minimize these uncertainties, developing a new design concept is a general research assignment. One of these concepts is the miniaturization of experimental setups. Investigating small-scale setups and projecting the relevant region of a column leads to more precise measurement data (4). Previous studies show various miniaturized setups which investigate the fluid dynamics in structured packings, resulting in reproducible and verifiable measurements (4, 5). Current studies at RUB show that miniaturized measuring cells are also applicable for the investigation of one-sided mass transfer and achieved reproducible results (6). Figure 1 shows the principle of this miniaturized measuring cell designed by RUB.

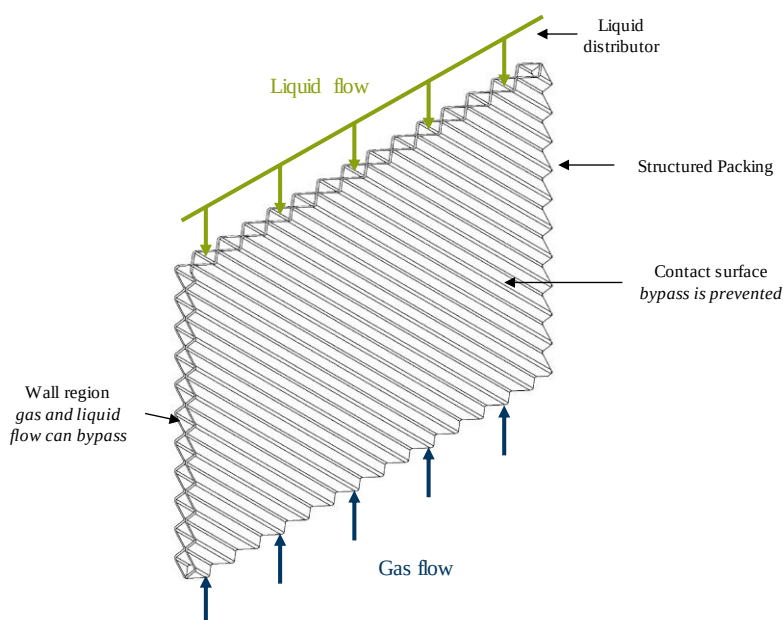


Fig. 1 Principle of the miniaturized measuring cell @RUB

At first it should be noted that designing the smallest possible experimental setup to reduce the required hold-up is one of the main aim. With a minimal amount of used mixture nearly every material system can be investigated, even expensive ones. This reduces the error in transferring standard test mixtures to industrial mixtures. A rectangular cross section is used to present the bulk region of a usual column. Similar to a usual column, gas and liquid are flowing countercurrent to one another. The main part consists of the structured packing with multiple packing sheets and elements, i.e. multiple packing layers on top of each other. To ensure a uniform liquid flow at the inlet, the liquid distributor consists of several dripping points – one for each packing channel. The contact between packing sheet and wall is sealed, so that no fluid flows between this gap and thus wall effects are prevented. The wall region perpendicular to the packing sheets has a predefined gap with a similar gap-to-packing ratio of an industrial-scale column. In the following, the mentioned measuring cell for one-sided mass transfer and a new measuring cell for two-sided mass transfer are presented.

2. Measuring cell with one-sided mass transfer

The measuring cell with one-sided mass transfer was developed within the research projects DFG: GR-206/11-1 and RE-1705/13-1 to improve the design method for columns with structured packings by investigating fluid dynamic and mass transfer of absorption and desorption phenomena. The detailed experimental setup bases on aspects described above is presented by Leuner et al. and Plate et al. (7, 8). These results lead to a profound understanding of the performance of miniaturized measuring cells, not only for the absorptive one. This includes measurements of the influence of the number of packing sheets on the fluid dynamics. Figure 2 a) shows the specific hold-up over the liquid load without a countercurrent gas flow. There is no dependency of the amount of packing sheets and the liquid hold-up. Aiming at a small scaled measuring cell with as less hold-up as possible, it is reasonable to install even two packing sheets.

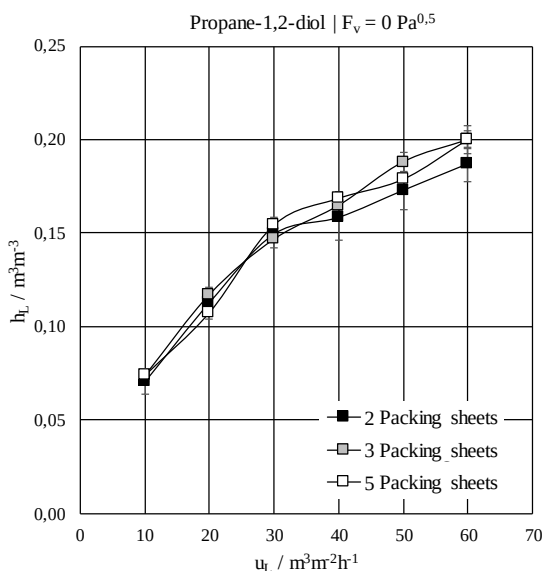


Fig. 2a) Specific hold-up for 2, 3 and 5 packing sheets and over the liquid load with propane-1,2-diol and the MONTZ-Pak B1.250.45 without countercurrent gas flow (8)

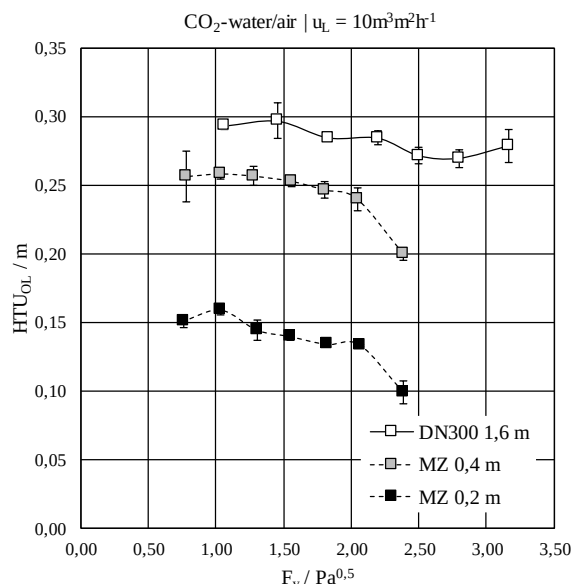


Fig. 2b) Overall HTU value over the gas load with CO₂-water/air for different setups: measuring cell with one and two packing elements, DN300-pilot-plant with 8 packing elements (6)

For mass transfer measurements the desorption of CO₂ from water is investigated. This standard test mixture is characterized by the fact that the gas-side mass transfer resistance is negligible. This study deals with the performance of the measuring cell with three packing sheets for different packing heights in comparison to a pilot-scale column with an inner diameter of 288 mm. Figure 2 b) illustrates the overall HTU value as a function of the gas load. It can be clearly seen that there is a dependency of the mass transfer on the number of packing elements. Installing two packing elements in the measuring cell results in an approximation to the performance of the DN300 column. The effects can be substantiated in different aspects. One possible explanation is that at higher bed heights the distance between the

bottom of the measuring cell and the packing outlet decreases. This can lead to a difference between the different heights, installed in the measuring cell. The deviation from the pilot-plant results can be justified with the ideal liquid distribution in the measuring cell and with the non-realistic installation of the packing elements. Because of the rectangular cross section, the packing elements in the measuring cell do not have 90°-offset. (6) However, there is a clearly dependency of the amount of packing elements and the mass transfer. A deeper investigation of these effects has to be performed.

Another aspect is the investigation of the wall region of the measuring cell. In previous studies the pressure drop and liquid hold-up with a variation of the gap width (3, 6 and 9 mm) was measured. Additionally, results without wall wipers are compared to a setup with installed wall wipers. The main observation is that there is no clear effect on these two settings when taking 3 and 6 mm into account. The 9 mm gap width shows a bypass effect of the gas, resulting in a lower pressure drop. But it should be noted that the ratio of the gap width to the whole cross section is bigger than in pilot-plants or even industrial-plants. Plate et al. and Stoter et al. mentioned another important effect concerning wetted wall wipers. Besides the effect of pouring the liquid phase back in the packing, wall wipers – especially wetted ones – also represent a resistance for the gas phase. (7, 5) This positive effect should be considered when designing a similar measuring cell.

3. Measuring cell with two-sided mass transfer

Within the cooperative research project ReProvAP the development of miniaturized measuring cells for investigating the fluid dynamic and mass transfer of distillation processes is advanced. Focusing on the determination of HETP values under different conditions, the results are discussed and compared to lab- and pilot-scaled plants which are also part of the project. Main requirement for the measuring cell is to minimize the used hold-up in comparison to a lab-scale column, so every material system can be measured even at an early stage of the design process.

Two new different experimental setups are developed by RUB and by the Technical University of Berlin (TUB). The concept is pictured in Figure 3. The measuring cell presented in this paper targets the height variation, the other one includes the length variation. By investigating various influential factors, the aim is to define one optimal miniaturized measuring cell that combines the findings of both setups. With this miniaturized measuring cell, the comparison to other columns and additional simulations of the performance of packed distillation columns with several dimensions it leads to a succeed scale-up procedure within the research project ReProvAP.

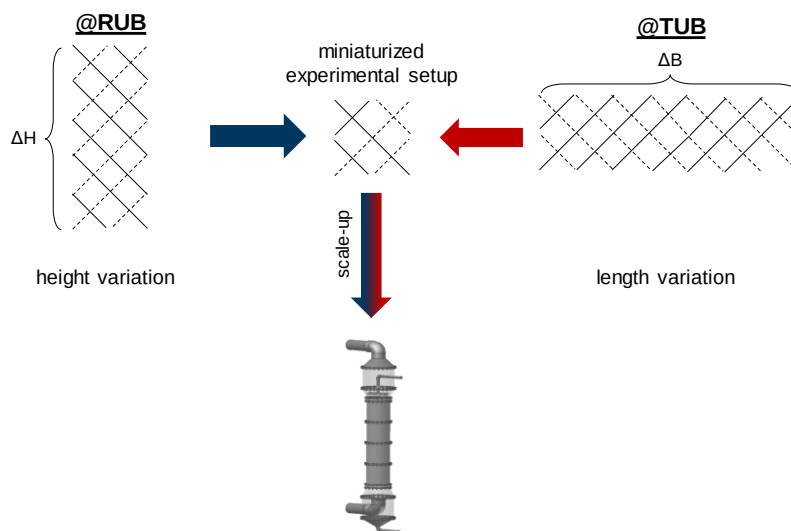


Fig. 3 Two different approaches to identify the optimal miniaturized experimental set-up for a new design method

3.1 Detailed Design

The general principle of the distillation measuring cell bases on the characteristics mentioned above (see Figure 1). The measuring cell is made of stainless steel. The main part are the two structured packing sheets with the dimensions of 100 x 100 mm which leads to a 106 x 6 mm free-cross section taking into account a 3 mm-distance between wall

and packing (wall region) and the seal between wall and packing (contact surface). With the requirement to utilize as less hold-up as possible it is reasonable to minimize the size of the measuring cell. A length of 100 mm leads to an equivalent DN30 column. Based on this, a height of 100 mm is chosen, so one packing element maps one entire continuous channel (see Figure 4).

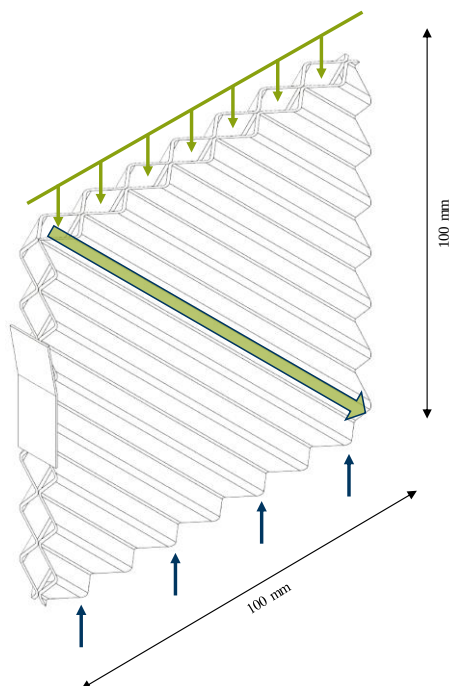


Fig. 4 Structured packing with two packing sheets and an area of 100x100 mm for the distillation measuring cell

Besides the separation section which contains the structured packing elements the measuring cell consists of four other sections: the bottom section, the sample-section, the liquid distributor and the top section (see Figure 5). To ensure that every kind of variation, e.g. packing type or material system, is feasible the measuring cell has to be modular.

Bottom-section

The bottom-section consists of a double-walled construction which acts as an internal evaporator. Thermal fluid flows through the outer gap and is heated by an external bath thermostat. The inner part is filled with the test mixture. For a sufficient mixing of the approx. 500 ml test mixture the inner wall is rough and boiling stones are added.

Sample-section

Oblique lances for sampling are installed right below the packing element. The attachment is optional, so measurements with one up to three lances at different positions are possible. The lances are located directly below a packing channel or between two channels. Superfluous sample is redirected in the bottom section.

Separation-section

In the separation-section the liquid and gas phase flow countercurrent over the packing element surface. To prevent wall effects, the longitudinal contact area to the wall is sealed with a wave-shaped teflon plate and a 0,5 mm FKM mat. The distance between packing and wall on the short side is 3 mm with installed wall wipers every 100 mm, so at least one wall wiper per packing element. To easily investigate the influence of the packing height every segment contains one packing element. There is no gap between the packing elements to avoid additional pressure drop, maldistribution and mass transfer at the edges of the packing elements. The elements are each rotated by 180° modelling a zigzag shape of the packing channels and thus returning the liquid to the packing.

Liquid distributor

For an ideal liquid distribution, the liquid distributor consists of one dripping point for each packing channel. This leads to approx. 11.700 dripping points per m². A uniform distribution is assumed due to a liquid feed on both sides of the liquid distributor. This has already been confirmed with the one-sided mass transfer measuring cell. However, during the proof of concept the dripping behavior will be investigated. When installing another packing type, a new

distributor-segment has to be manufactured because of the different channel length and thus the pipe position in the longitudinal direction is different.

Top-section

In the top-section the gas phase flows from the inner part of the measuring cell to the external total condenser and the reflux-section. To avoid unintentional evaporation in the pipelines, the reflux has to be heated up near boiling point so that the temperature profile inside the cell is not negatively affected. (9)

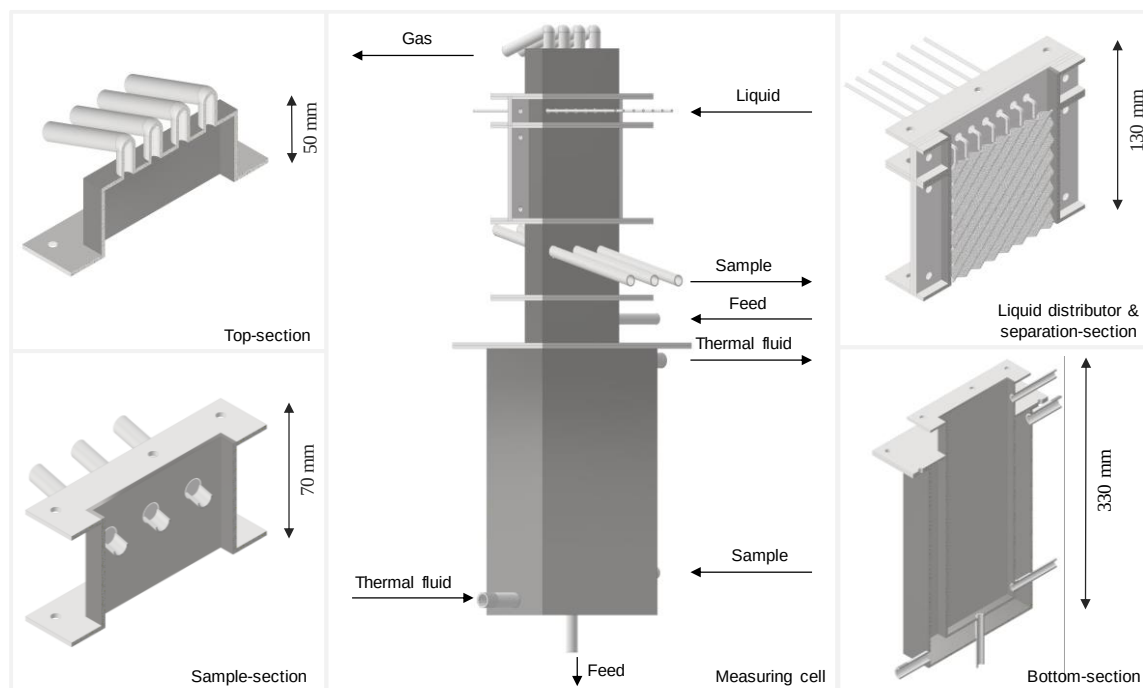


Fig. 5 3D-Model of the measuring cell developed at RUB

3.2 Experimental procedure and conditions

First, measurements with the test mixture Cyclohexane/n-Heptane (C6/C7) are performed. This material system is a common recommended test mixture to investigate distillation-phenomena (10). Two sheets of standard structured packing are inserted. They are an unperforated version supplied by Sulzer Chemtech AG with similar dimensions as the structured packing MellapakTM 500.Y of the same company. The experiments are performed at total reflux with a pressure round about 1 bar and at a load range up to the flooding point. During the experiments the pressure drop over the packing element, the overall pressure, the top and bottom temperature, the reflux flow rate and the top and bottom mixture concentration are measured. The gas and liquid loads are related to the free flow area. After investigating the start-setup, several setup combinations will follow (see. Table 1).

Table 1. Measurement plan for the distillation measuring cell @RUB

Packing height	Test mixture
1 x 100 mm	Ideal-Benchmark: C6/C7
2 x 100 mm	Aqueous
4 x 100 mm	Wide-boiling
1 x 200 mm	Viscous
2 x 200 mm	

The dependency of the packing height and the amount of packing elements on the performance is tested with two different element heights: 100 mm and 200 mm. In addition to the benchmark-system C6/C7, other material systems with aqueous, wide-boiling and viscous properties are used.

For a first verification the results are compared to the measuring cell from TUB (see Figure 3). The next step will be a classification with other scaled experimental setups. For this, several plants starting from DN50 up to DN250 are investigated within the research project ReProvAP. Although most of the constructional aspects of the measuring cell are designed similar to larger scaled distillation columns, naming the wall regions or the liquid distribution, small packing heights can lead to a deviation of the separation performance. In order to evaluate those effects, various height will be investigated. All in all, it can be assumed that the performance of the measuring cell is comparable with the performance of larger scaled columns which shows Figure 2b).

Additional simulation results will inform about the detailed phenomenological findings to improve the measuring cell-concepts. By varying the setup, the sensitivity of the HETP value to the different influencing factors will be determined which enables the development of a new scale-up procedure.

3. Conclusion

To develop a more accurate design method for structured packing distillation columns, a concept for a new miniaturized measuring cell is presented. Experimental investigations with this setup will reduce uncertainties in the predictability of the industrial column efficiency. First, the general concept of miniaturized setups at Ruhr University Bochum is described. To represent the bulk region of an industrial column, a rectangular cross section with several packing sheets is recommended. For an ideal liquid distribution one dripping point for each packing channel is installed. Based on this basis-concept, a measuring cell for absorption was developed and investigated. With this background information the distillation measuring cell is developed. The setup consists of a free-cross section of 106 x 6 mm with two packing sheets and several packing elements to determine the influence of the height on the mass transfer. Besides the variation of the packing type, the test mixture is also modified. These investigations lead to a data set of HETP values for various setup and mixture combinations. With a comparison between the two measuring cells from RUB and TUB, the optimal measuring cell design can be found. Including the different scaled plants and the simulation studies within the research project ReProvAP, a new design method can be developed and proved.

Acknowledgements

Special thanks got to the Federal Ministry of Education and Research, the Sulzer AG for the provision of the packing and all the project partners for their support.

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