Improved and Innovative Methods for the Characterization of Random and Structured Packings

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Design and scale-up of packed absorption columns are based on experimental data from pilot columns and on expert knowledge as well as experience of the packing manufacturers. The characterization of column internals itself depends on the respective test facility, where the experiments are carried out (Schultes, 2013). In contrast to columns in industrial scale, smaller ones are significantly affected by wall flow, which is a major reason for parameter uncertainties. To overcome these uncertainties in the design procedure of packed columns, three approaches are suggested. One is based on an improved characterization of maldistribution by the use of a wire-mesh-sensor. It is reviewed, that the sensor can be flexibly used by simple investigation of the distribution of the measured phase fractions and to determine dynamics during column operation. Furthermore, more detailed knowledge about axial and radial spreading of the liquid can be obtained by residence time distribution experiments. The two other approaches are the development of new characterization methods: A miniaturized experimental setup and a newly developed model. The miniaturized experimental setup is based on the idea of eliminating the influence of end effects for structured packings. The new model approach considers large scale maldistributions, instationary hold-up and mass transfer.

1. Introduction

Research in the field of mass transfer columns has more than 100 years of history. Nevertheless, there are many research areas that are not exhausted and still need precise examination. In 2013 Schultes (2013) formulated and discussed several research topics in the field of mass transfer columns from the industrial point of view. To name just the relevant topics in this work, these are standardization of equipment and experimental procedures used in internal characterization and progress in model development for predictive description, both with regard to fluid dynamics and separation efficiency.

In the last decades several groups already treated the topic of standardization successfully, establishing recommendations to efficient and comparable column internal characterization (Górak and Grünewald, 2013; Hoffmann et al., 2007; Kunze et al., 2012; Kunze et al., 2015; Rejl et al., 2009; Wolf et al., 2015). A comprehensive overview of their results can be found in Hegely et al. (2017). However, to minimize influences by end effects and maldistribution, comparable large column diameters are necessary, increasing the effort and cost for the experimental characterization (Hegely et al., 2017).

This contribution is dedicated to reduce the effort by improving conventional characterization methods with new measurement techniques to monitor the flow distribution, on the one hand. On the other hand, by introducing new methods to characterize column internals that are an innovative predictive cell model and a miniaturized experimental setup. Both are based on the idea that the internal characteristics can be reduced to a small representative volume with selective consideration of non-ideal effects during column operation.

2. Characterization and Design of Packed Columns - State of Art

The characterization of column internals is usually performed with standardized systems to determine the specific pressure drop, the liquid hold-up, the overall liquid and gas site mass transfer and the effective phase interface for an internal. Usually, cross-sectional averaged, integral quantities are used. The transfer of the

characteristic parameters of standardized systems to the industrial relevant systems is based on specific models and expert knowledge of the packing manufacturer.

A common problem of the internal characterization is the uncertainty of the determined parameters that can deviate significantly depending on the test facility (Schultes, 2013). In particular, wall flow and cross sectional mixing are the main effects resulting in deviations from ideal distribution of the liquid phase. Although standardized characterization measurements minimize these uncertainties, test facilities beyond a column diameter of ten times the characteristic random packing length (Kouri and Sohlo, 1996) or 0.5 m column diameter for structured packings (Olujic, 1999) are necessary to reduce wall effects to an acceptable magnitude. Nevertheless, smaller columns could be used for characterization, but considerable expertise is needed to subtract the influence of wall flow.

3. Approaches for an Improved Method for Characterization of Packings

3.1 Application of Cross-Sectional High Resolution Sensor Technique

Liquid phase maldistribution, especially wall flow, has a significant contribution to the uncertainties of mass transfer parameters determined by state of the art characterization. Conversely, the detailed knowledge of wall flow and its development can be used to determine the influence on the determination of mass transfer parameters. Possibilities to characterize the wall flow are particularly liquid collectors and non-invasive tomographic systems. However, the former leads to complex experimental setups due to its invasive nature and has a low spatial resolution. For the latter, the setup is not less complex and advanced knowledge on image reconstruction is necessary to extract reliable information from the measured data.

Since liquid phase fractions can be determined with high time (up to 10,000 frames per second) and spatial resolutions (determined by the distance between two wires in a plane), the wire-mesh sensor is able to determine local liquid maldistributions and dynamics of the liquid flow (Schubert et al., 2011). It was introduced in 1998 by Prasser et al. (1998) as low invasive tomographic system for gas-liquid flows.

The sensor consists of two orthogonal planes of wires, one plane being used as transmitter and the other as receiver. It can be operated in electrical conductivity or permittivity mode to determine the phase fractions at each crossing point between the wires of both planes (Schubert et al., 2011). The wire-mesh sensor is calibrated with the process fluids, e.g. pure water and air. The sensor output signal can then directly be transferred to the phase fractions.

The following three paragraphs are intended to give a brief overview of the applicability of the wire-mesh sensor for the investigation of column performance. While measurements of phase fractions and dynamic features were already investigated by Grünewald et al. (2011) and Schubert et al. (2011), respectively, the applicability to determine residence time distributions in packed columns is a new approach, currently investigated.

Determination of Phase Fraction Distribution

For the measurement of integral phase fraction distribution, the wire-mesh sensor is placed 10 to 20 cm below the packing. Figure 1a shows a time averaged liquid phase fraction over the x and y grid directions of the sensor for a structured packing at a gas load of 3.3 $Pa^{0.5}$ (32x32 sensor). Even this simple representation makes it possible to identify regions of elevated irrigation. By analysing the cumulated phase fractions of a ring close to the wall, one can compare the wall flow of one packing with another at the same gas loads, since the gas velocity slightly influences the measured phase fractions. This was already introduced by Grünewald et al. (2011) and is useful for the characterization and the development of random and structured packings as it makes the influence of maldistribution comparable to well investigated internals, e.g. the Pall Ring 25.

Column Operation Dynamics

Another useful application of the wire-mesh sensor is the knowledge of dynamic features during column operation. Figure 1b shows the cross sectional sum of measured phase fractions over time for a change in the liquid load from 10 to 20 $m^3 \cdot m^{-2} \cdot h^{-1}$. These measurements allow to determine the fluid dynamic response time of a column internal, at a specific packed height, with operational changes, considering the response time of the rest of the plant. This could be useful for the control of plants with varying gas or liquid loads, e.g. in flue gas purification. Moreover, it could be used to monitor stationary fluid dynamic conditions during characterization measurements.
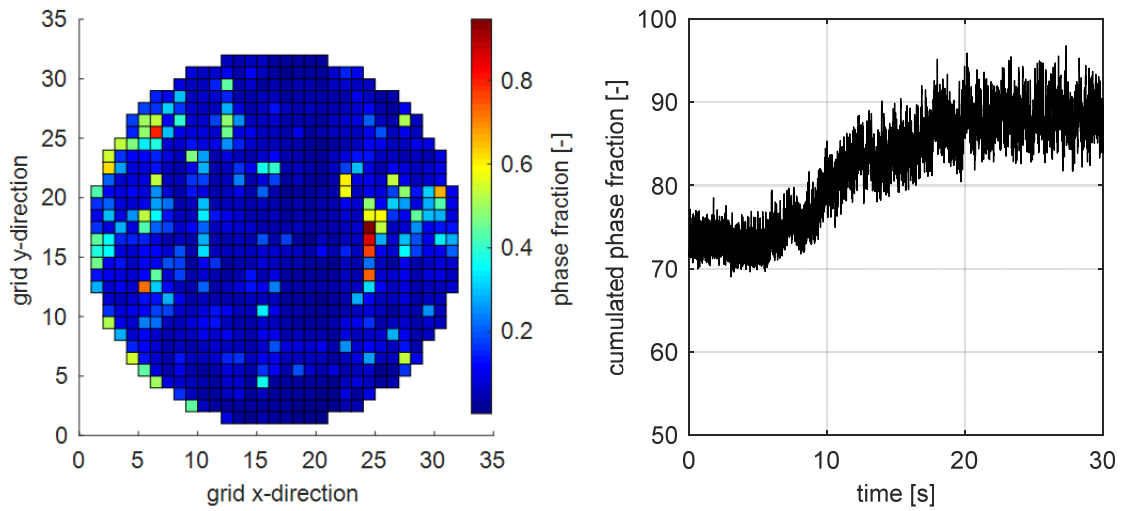
Figure 1: (a) Phase fraction distribution of a structured packing above the loading point, (b) Dynamic response of changed liquid load for a structured packing

Determination of Residence Time Distribution

For more detailed information about the distribution and especially its time dependence, residence time experiments with an inert tracer are common practice. Measuring the conductivity with the wire-mesh sensor, sodium chloride was found to be a suitable tracer. With a pulse injection in the center of the column by syringe or a vessel of defined volume, it is possible to identify radial and axial mixing properties. By injection on the column wall, it is possible to determine average wall flow velocities and the fraction of tracer redistributed from the wall to the bulk of the packing. Figure 2a shows the time accumulated local response of the tracer experiments over the column cross section, measured by a 64x64 wire-mesh sensor. Figure 2b presents the normalized residence time distribution of detected tracer on the whole cross section of the column. The displayed results are for a central pulse injection in a bed with a height of 0.5 m of Pall Rings 25 and a column diameter of 288 mm. The liquid load is fixed at 10 $m^3 \cdot m^{-2} \cdot h^{-1}$ without countercurrent gas flow.
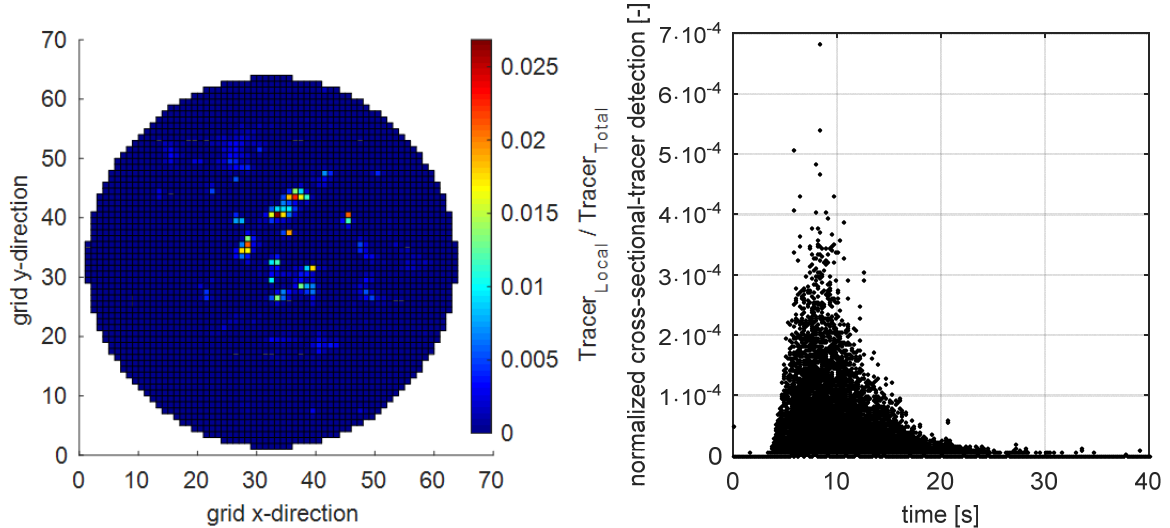

Figure 2: Central impulse injection (a) Normalized spatial tracer detection, (b) Normalized residence time distribution

In this case, the packed height was chosen as 0.5 m to ensure absence of the tracer from the column wall, which is ideal for the determination of radial mixing. A few strong signals close to the column center indicate regions of lower radial mixing. Close to the wall the signals of detected tracer are weaker but occur more frequently. This indicates that the tracer is affected by increased mixing, which is lowering the signal but

increasing the number of liquid trickles containing tracer. To gain knowledge about the axial mixing, the results of Figure 2b can be used. After first tracer detection there is a rapid increase followed by a peak and a tailed decrease of the detected tracer. The high time resolution up to 10,000 Hz and the exclusion of end effects below the packing are adequate conditions for the determination of axial mixing. These results may help for a qualitatively assessment of mass transfer equipment especially in case of complex mass transfer assessment, due to thermo-physical properties.

3.2 Miniaturized Experimental Set-Up

The basic idea of this approach is to reduce the dimensions of the experimental set-up to a minimum required size that allows representative measurements of fluid dynamic and mass transfer characteristics as predominant in industrial sized columns. Especially the periodic geometry of structured packings is suitable for reducing the complexity to a small section of a packed column and to investigate all necessary parameters for column design in a miniaturized experimental set-up.

This set-up comprises different miniaturized measuring cells for the investigation of the packing characteristics with little effort and minimum amount of substances on a laboratory scale. Beside cost savings, this approach enables investigations of packing material and geometry at a very early stage of design (Repke et al., 2011). Furthermore, the apparatuses are very flexible and accessible, providing relieved insight into fluid dynamics and mass transfer conditions of industrial relevant and potentially hazardous fluids.

In order to analyze the connection between a miniaturized experimental set-up and a column equipped with a structured packing, a measuring cell has been built (see Figure 3a). The cell enables to determine fluid dynamic parameters like liquid hold-up and pressure drop under ideal boundary conditions. The experimental set-up consists of a rectangular column with various numbers of packings sheets. To minimize end effects, normally occurring in technical columns, several design aspects have been considered. The liquid distributor as well as the gas distributor are designed for a uniform distribution directly in the inlet area of the packing. Furthermore, the gap between the outer packing sheets and the wall is filled with silicon negatives to prevent wall effects at the longitudinal side.

Figure 3b illustrates results of the specific hold-up of propane-1,2-diol as a function of the liquid load for two, three and five packing sheets of MONTZ-Pak B1-250.45. The hold-up was measured using the draining method, correcting the results with measurements of the empty column. To ensure fully wetted packing sheets the liquid propane-1,2-diol has been used. The results show that the specific hold-up of the different numbers of packing sheets are in good agreement and follow the typical characteristic curve of holdups as reported in literature. This simple investigation indicates that it might be possible to reduce the investigated volume to a minimum and is gradually developed. Henceforth, the results determined in the miniaturized set-up should be implemented in a numerical cell model to design an industrial sized, packed column. The miniaturized approach is currently being pursued in cooperation with the Technical University of Berlin.
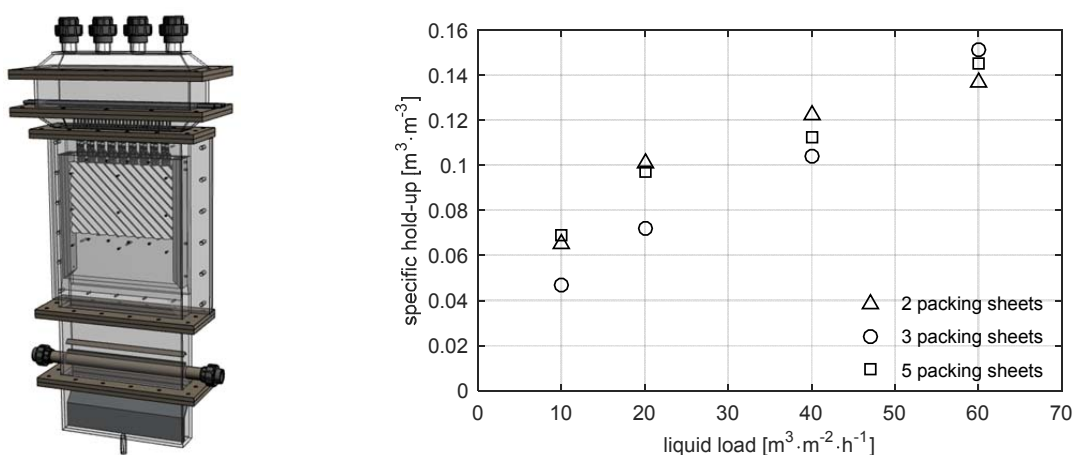

Figure 3: (a) Measurement cell with various numbers of packing sheets, (b) Specific hold-up for different numbers of packing sheets and liquid load on MONTZ-Pak B1-250.45

3.3 Model Approach for Improved Scale-Up

Maldistribution is one of the major challenges for the design and scale-up of small to large column dimensions. Considering only large scale maldistributions, wall flow is the most important quantity. It is known how to design a liquid redistributor, e.g. by Bartlok (2002), for certain amounts of wall flow, but the height after it is

necessary to redistribute is difficult to determine. In industrial praxis, the influence of wall flow on the mass transfer can be described by two-column models, which predict the reduction of mass transfer satisfactory. However, two-column models are unable to describe the development of wall flow along the height of the column until equilibrium flow is reached (Albright, 1984). Due to the ability to describe large model volumes, dispersion or cell models (e.g. series of stirred tank reactors) have usually been used in the past to simulate whole columns. Previous model parameters for wall flow development have been empirical fitted to results of a specific column diameter, which cannot be easily transferred to other diameters. In order to consider the influence of the column diameter, a new model approach was developed. The main ideas of the model are:

- Wall flow evaluation based on the distribution of a single random packing to be geometrical independent.
- Differentiation of the packed column in bulk and wall regions with two different distribution parameters.
- The distribution parameters can be fitted by residence time distribution experiments with an inert tracer.
- The definition of the local quantities hold-up, concentration and effective mass transfer area for each cell volume.
- An ideal mixed cell volume.

First, the model was used to predict wall flow quantity and development over the packed height. The necessary model parameters to be determined were the liquid spreading factors for bulk and wall regions. These parameters were obtained by experimental residence time distribution experiments on the wall and in the bulk of the column using a wire-mesh sensor as presented in Figure 2a. Figure 4a shows the model geometry with the assumed net distribution mechanism for a single random packing (Figure 4b). The simulation results of the model, which are presented in Figure 4c, are in good agreement with the measured wall flow by Kouri and Sohlo (1996).
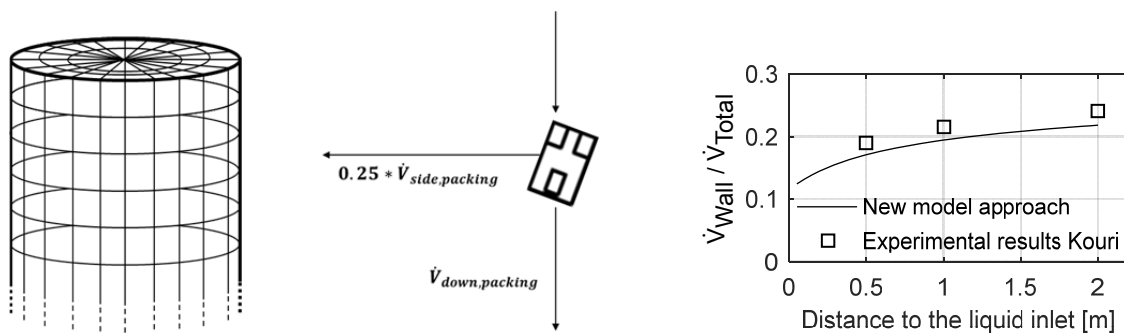

Figure 4: (a) Geometry of the overall model, (b) Definition of the net flows out of a single packing, (c) Comparison of the simulated wall flow to experimental results

4. Conclusion

The improvement of column internal characterization was presented by means of three approaches. First, it is suggested to expand standardization by characterization of the maldistribution. Second, a new miniaturized experimental setup was introduced for the improved determination of fluid dynamic and mass transfer coefficients. Third, an innovative model approach is suggested that simplifies the consideration of large scale maldistributions.

For the characterization of liquid maldistribution, the wire-mesh sensor is a simple and flexible technology with various applications. The sensors output signal, the phase fractions, can directly be used to compare distributions of different packings at constant liquid and gas loads. Moreover, column operation dynamics, e.g. operational changes or times between two steady states, can be monitored by the cross sectional sum of phase fractions. Another advantage of the wire-mesh sensor is the possibility to determine the residence time distribution of the liquid flow within the column. The experimental distributions can be used to determine radial and axial mixing properties and its deviations from plug flow. On the basis of this, qualitatively comparisons of different internals and packed heights are possible.

The introduced new approach for the characterization of fluid dynamic and mass transfer coefficients is based on the concept of an idealized and miniaturized experimental setup. The aim of the laboratory sized setup is the realization of a representative bulk cell of an industrial sized column. Therefore, the design excludes end

effects occurring under usual operational conditions. The first results for the determination of the specific hold-up are promising as the number of investigated packing sheets only had small influence.

The fundamental idea of the new model approach is the distinction into representative bulk and wall cells that is realized by means of two distribution parameters. One of the biggest advantages of the approach is the determination of wall flow quantity and development. The parameters can be determined by wire-mesh sensor distribution measurements. The structure of the model allows further implementation of mass transfer and small-scale maldistribution at simultaneously large simulation volumes.

Aim of future investigations is the connection of the featured methods, e.g. the transfer of the results from the miniaturized setup to the numerical cell model for simplified scale-up or the use of wire-mesh sensor data for the determination of wall behavior that can be implemented into the model or directly used in two column models together with the ideal bulk data of the miniaturized setup. Moreover, the suitability of the wire-mesh sensor and the miniaturized setup for different fluid systems, e.g. organics, will be tested.

Acknowledgments

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