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An Advanced Approach to Design Structured Packing Columns using Miniaturized Experimental Set-Ups

Hannes Leuner[a,*], Mark Hapke[b], Johannes Sacher[a], Marcus Grünewald[b], Jens-Uwe Repke[a]

[a]Technical University of Berlin, Process Dynamics and Operations Group, Str. des 17. Juni 135, 10623 Berlin, Germany
[b]Ruhr-University Bochum, Laboratory of Fluid Separations, Universitätsstraße 150, 44801 Bochum, Germany
h.leuner@tu-berlin.de

An advanced design approach for structured packing columns is presented that includes experimental investigations in a miniaturized set-up, which are intended to be combined with a numerical cell-model. First tests to define a suitable miniaturized set-up are discussed for two different novel experimental set-ups that investigate either the influence of the number of packing sheets or the geometry of a packing sheet focusing on liquid hold-up values for MONTZ-Pak B1-250.45 and propane-1,2-diol. Liquid hold-up values are compared to existing hold-up correlations from the literature.

1. Introduction

Structured packing columns are widely used for distillation and absorption processes in the chemical industry. This packing type ensures fluid separations on a higher efficiency level including lower gas pressure losses than other systems due to their unique geometry. However, current design methods for these columns base on simplified and rough assumptions about fluid dynamics and mass transfer that are not able to predict actual conditions inside the columns with a sufficient accuracy or need to be extensively determined within costly and time-consuming pilot plant investigations. This is even more crucial in case of unknown or complex multi-component mixtures. Hence, structured packing columns are nowadays usually designed using overdesign factors, which leads to oversized equipment as well as higher investment- and operational costs. To reduce such uncertainties and time requirements, sophisticated design methods are needed that predict column performance more accurately in a reduced time (Schultes, 2013, Repke et al., 2011, Kenig, 2011).
Many of the established design methods for structured packing columns rely on the plug-flow assumption of uniform conditions across the cross-sectional area of a column, but actually small- and large-scale maldistributions occur inherently along and across a column. Furthermore, mass transfer correlations depend on semi-empirical models like Billet&Schultes (1993), Billet&Schultes (1999) and Olujic et al. (2014) based on simplified, often binary mixtures that do not represent real fluid-systems inside columns (Schultes, 2013).
This work presents an alternative design approach for structured packing columns, which is currently being developed in a cooperative research project. The basic idea is to connect experimental investigations in a miniaturized set-up with a numerical cell-model to be able to design a whole column and overcome the uncertainties of conventional design methods. The miniaturized experimental set-up is therefore a representative cutout section from a structured packing column that is investigated to achieve insight on fluid dynamics and mass transfer conditions of the real fluid system on the real packing material at a very early stage of the design process. The cell-model itself subdivides a real scale column in segments across the length and the cross section of the column and solves mass and energy balances along the major paths including a parametrization with the experimental results of the miniaturized set-up (Van Holt&Grünewald, 2016). That procedure intends to increase the accuracy of column performance predictions and replaces costly and time-consuming pilot plant investigations. Furthermore, the investigations in the miniaturized set-ups provide deeper insight to fluid dynamic and mass transfer processes in structured packing columns especially in case of complex multi-component mixtures.

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The research project comprises three major steps at which the first step is presented in this paper including the design of a suitable miniaturized experimental set-up to perform representative measurements of fluid dynamics and mass transfer characteristics. In the second step, a numerical cell-model for structured packing columns needs to be developed, which considers the findings from the miniaturized experimental set-up. In the third step, both results need to be combined to an advanced design approach for structured packings that is meant to predict real scale column performance.

2. First step - Miniaturized Experimental set-ups

The miniaturized experimental set-up should allow measurements of liquid hold-up, gas pressure drop and mass transfer coefficients in an environment that is on the one hand large enough to represent real separation fluid behavior like in a column and on the other hand as small as possible to reduce complexity and costs. Therefore, two closely aligned miniaturized set-ups were built to investigate either the influence of the number of packing sheet (by Ruhr-University Bochum) or the influence of the geometry of the packing sheet (by Technical University of Berlin) to find the minimum required dimension for such a miniaturized set-up. In the first set-up (left side in Figure 1), fluid dynamic parameters like liquid hold-up and gas pressure loss were measured for different quantities of packing sheets starting at a high number towards a small number of packing sheets to analyze the connection between a miniaturized set-up to a structured packing column. In the second set-up (right side in Figure 1), film thickness measurements were performed on a single packing sheet starting at a flat, textured packing sheet and continuing towards a real packing sheet by a stepwise incensement of the complexity of the geometry.

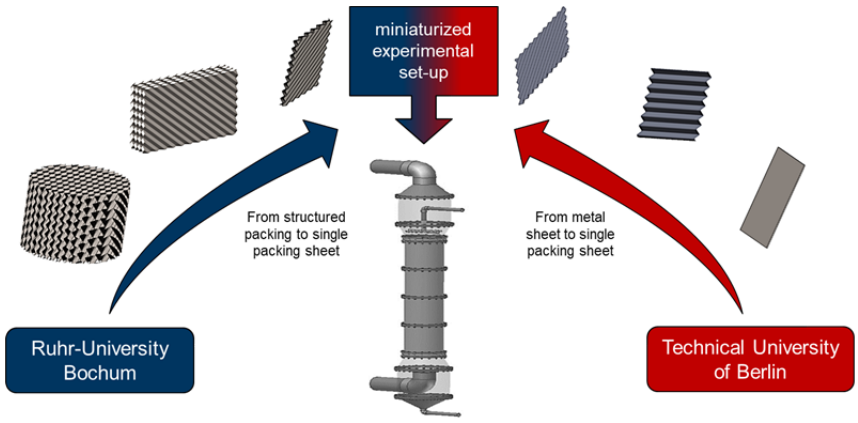

Figure 1: Road map to design the miniaturized experimental set-up.

Both set-ups use MONTZ-Pak B1-250.45 packing material and a similar sensor set-up to ensure comparability. The research project aims preliminary on the development of the design method focusing on the film flow phenomena and fully wetted packing sheets, which requires a working liquid with a low surface tension. To ensure a scientifically justified comparability of the measurement results of both experimental set-ups and prevent mixing errors, a one liquid component system is necessary to select. Hence, a propane-1,2-diol/air system ($\sigma_L = 30.56\ mN/m$, $\eta_L = 55\ mPas$) has been chosen that ensures film flow conditions and fully wetted packing sheets.

2.1 Miniaturized experimental set-up to investigate the number of packing sheets

The first experimental set-up is a rectangular column with a variable number of packing sheets and cross-section up to $357\ x\ 60\ mm$ (see Figure 2). The liquid distributor at the top of the measurement set-up consists of as many dripping points as packings channels exists in the particular case and has been specially designed for an even liquid flow distribution. Each dripping point feeds one packing channel with liquid to realize a uniform liquid distribution already at the inlet of the packing. Furthermore, the gas flow system was designed for a uniform gas distribution. The gap between the outer packing sheets and the wall at the longitudinal side is sealed with silicon to prevent wall effects. The liquid hold-up was measured using the draining method that is considered to represent the dynamic liquid hold-up. The results were corrected by measurements of an empty column to take the hold-up of the liquid distributor and the liquid of the free volume into account. Additionally, the gas phase pressure drop between inlet and outlet of the structured packing as well as the liquid distribution of a tracer marked flow can be measured. Figure 3 illustrates the results of the dynamic liquid hold-up as a function of the liquid load without a countercurrent gas flow for two, three and five installed

packing sheets. The results for the different number of packing sheets are in good agreement for each investigated liquid load, which indicates that the dynamic liquid hold-up without a counter current gas flow is independent of the number of packing sheets. The dynamic liquid hold-up results for three packing sheets, which deviate slightly from the curve characteristic, are within the error tolerance and increases for higher liquid loads up to a value of $0.15\ m^3/m^3$.

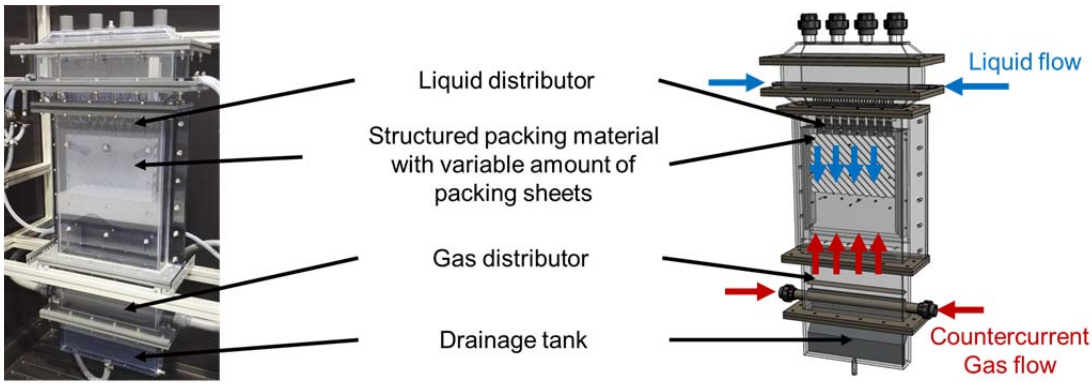

Figure 2: Miniaturized experimental set-up to investigate the influence of the number of packings sheets with the draining method.

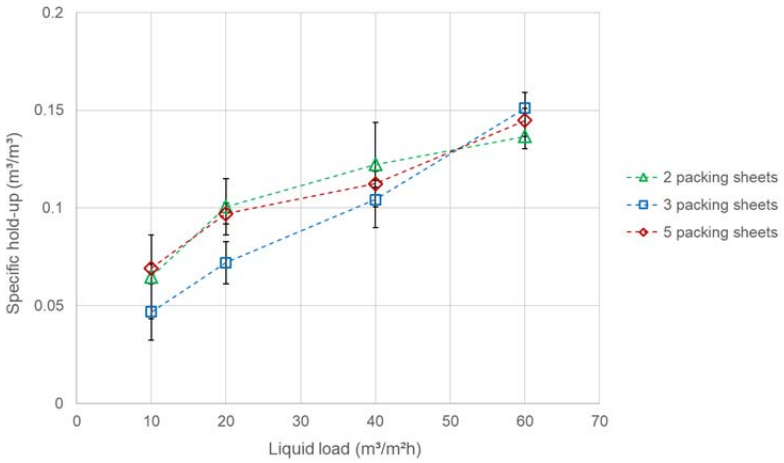

Figure 3: Dynamic liquid hold-up for different numbers of packing sheets and liquid loads of propane-1,2-diol on MONTZ-Pak B1.250.45.

2.2 Miniaturized experimental set-up to investigate the geometry of packing sheets

Besides the experimental set-up to investigate the influence of the number of packing sheets, a second experimental set-up has been designed where single packing sheet ($230x700mm$) of different geometries can be investigated (see Figure 4). The liquid enters the packing material on the top of the set-up through a liquid distributor system including a small gap to ensure stable and uniformly distributed film flow conditions. The gas flow system is as well designed to ensure an even gas flow distribution.

Within this set-up, film thickness measurements using the Light-Induced Fluorescence (LIF)-Method were performed with a high spatial resolution. Therefore, a fluorescent dye (Rhodamine B) is added to the liquid with a concentration of $5\ mg/l$. The intensity of the fluorescent light ($\lambda_{RhB} > 570nm$) corresponds to the local film thickness on the packing material which is recorded with a scientific CCD-Camera (PCO.2000). A green LED light ($\lambda_{LED} = 500 - 540nm$, Lumimax LQ100) is used to excite the fluorescent dye. To link the recorded light intensities of the fluorescent light to film thickness values, a calibration is performed including a wedge cuvette and a spatial normalization of the excitation light.

Figure 5 illustrates the results of the mean measured film thickness for laminar flow on a flat metal plate including the MONTZ-Pak B1 microstructure at different positive and negative inclination angles α referring to a vertical plane. Furthermore, the figure contains the theoretical estimated film thickness for laminar film flow on smooth surfaces according to Nusselt (1916). The experimental results and the solution of the Nusselt increase both with higher liquid loads on the sheet. For liquid load values above $20\ m^3/m^2h$, the measured mean film thickness

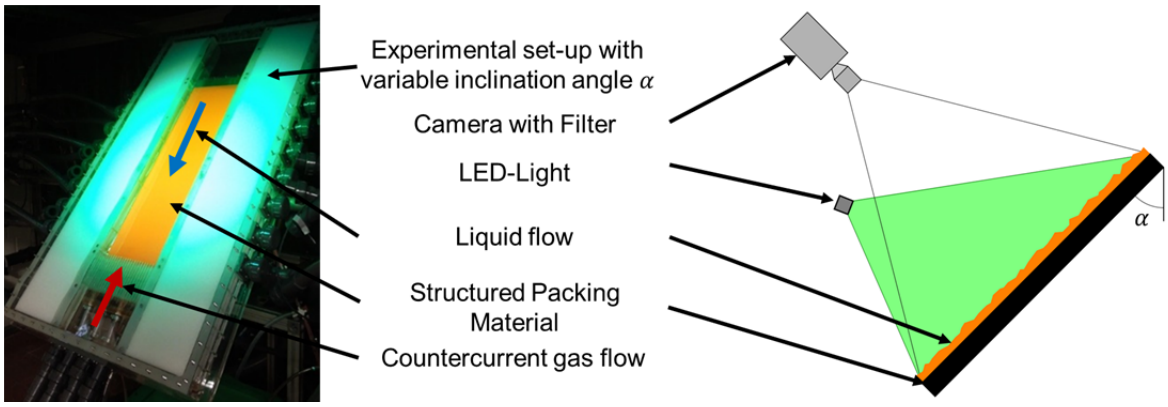

Figure 4: Miniaturized experimental set-up to investigate the geometry of packing sheets with the Light-Induced Fluorescence Method (LIF).

on the microstructure are higher than the Nusselt solution for smooth surfaces independently from the inclination angle for positive inclined angles (Figure 5 (a)). The experimental results and the solution of the Nusselt increase both with higher liquid loads on the sheet. For liquid load values above $20\ m^3/m^2h$, the measured mean film thicknesses on the microstructure are higher than the Nusselt solution for smooth surfaces independently from the inclination angle for positive inclined angles (Figure 5 (a)). That leads to $0.2 - 6\%$ higher film thickness values for the MONTZ-Pak B1 microstructure compared to a smooth plate. Film thicknesses on negative inclined angles tend to be rather smaller with an increasing absolute value of the inclination angle (Figure 5 (b)). Film thickness values at $10\ m^3/m^2h$ were recorded in a not fully wetted environment, which does not represent the film flow.

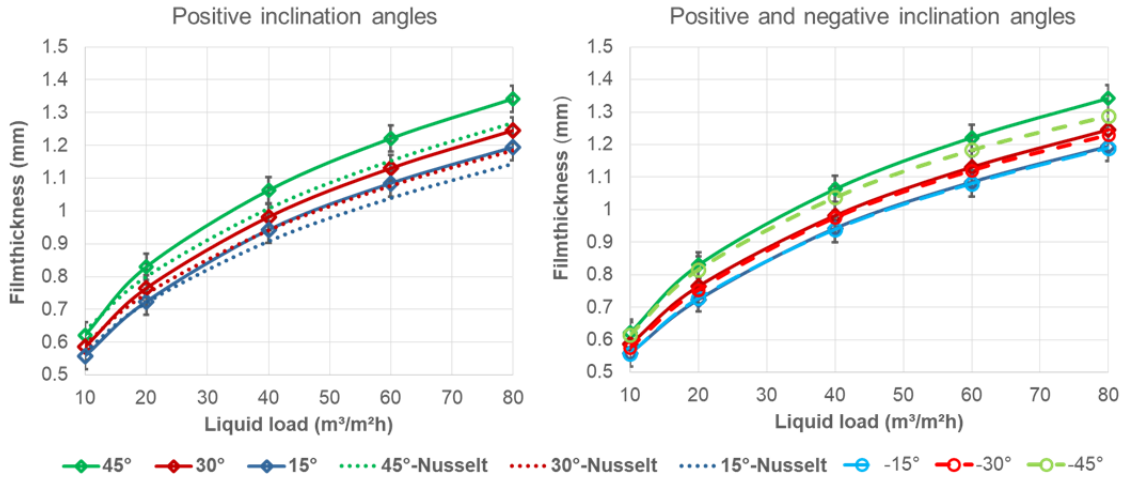

Figure 5: Mean film thicknesses at positive (a) and negative (b) inclination angles for laminar flow of propane-1,2-diol without counter current gas flow on a flat plate with MONTZ-Pak B1 microstructure.

To calculate the total liquid hold-up, the film thickness results from the flat, microstructured plate were further implemented in a geometrical scale-up model to calculate describing the shape of a real macrostructured packing sheet, which is presented more in detail in Leuner et al. (2018). This model includes a discretization of the real packing sheet into small quadratic areas based on the input of geometrical dimensions such as amplitude of macrostructure, wave to wave distance and curvature radius (see detail on Figure 6). Each area is assumed to be a 3D-orientated regular flat plate with a certain inclination angle that is fully wetted by a liquid film flow. The liquid film flow direction on that area is defined by the force of gravity. Depending on the orientation of the area inside the 3D-space, the force of gravity affects the liquid flow at a certain angle named effective liquid flow angle, which is calculated for every area (see Figure 6). The effective liquid flow angle directly influences the film thickness in the corresponding area at which the value of the effective liquid flow angle can be either positive or negative depending on the position of the area on the macrostructure of packing sheet. The corresponding film thickness for each area A_i and liquid load B_j is taken from the experimental results shown in figure 5 by 2D interpolating between positive and negative inclination angles α_i from the measurement set-up

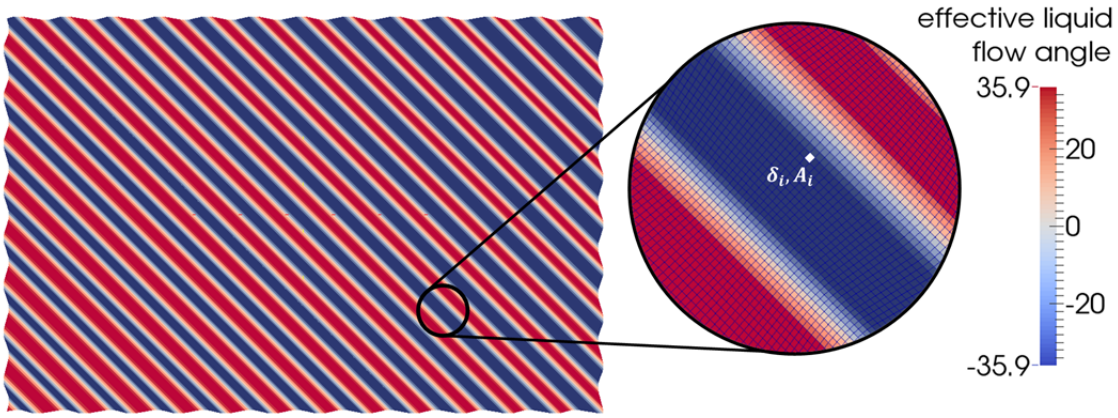

Figure 6: Discretization of single packing sheet and effective liquid flow angle determination for MONTZ-Pak B1-250.45.

and liquid loads B_j by assuming a constant liquid load across the packing sheet. The volume-specific liquid hold-up $h_{L,j}$ on the packing sheet is further calculated by summation of the film thickness of every area A_i.

2.3 Comparison of experimental results to existing hold-up correlations

The results of the liquid hold-ups for both measurement set-ups are compared to existing analytical hold-up correlations of Billet&Schultes (1993) and the delft-model of Olujic et al. (2004) for the total liquid hold-up and the semi-empirical correlation of Billet&Schultes (1999) for the dynamic liquid hold-up (see Figure 7).

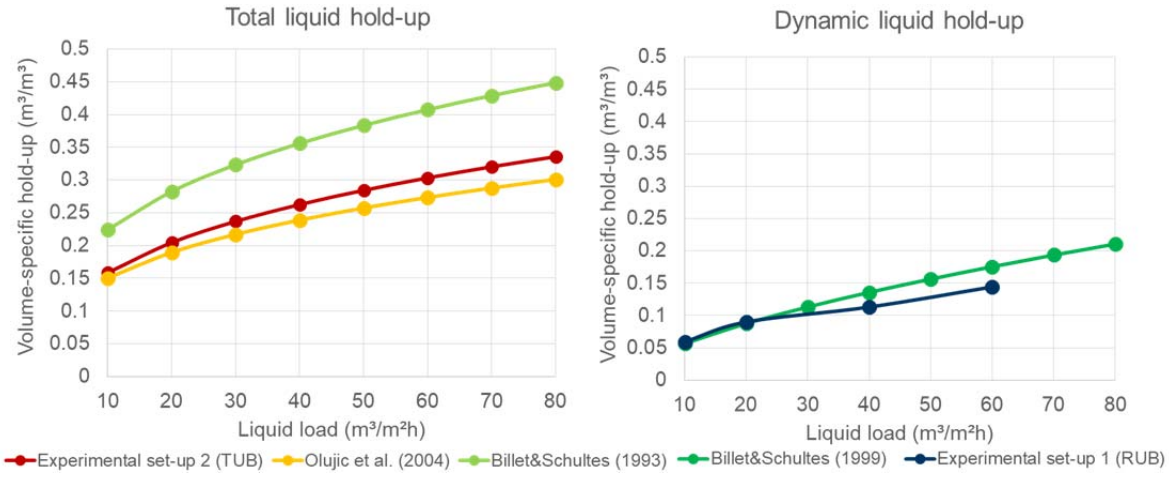

Figure 7: Volume-specific hold-up measurements and correlations for (a) the total liquid hold-up and (b) dynamic liquid hold up of propane-1,2-diol for MONTZ-Pak B1.250.45 on film flow conditions.

The total liquid hold-up determined by the second experimental set-up (TUB) is only $5 - 12\%$ higher than the results of the delft-model (Olujic et al., 2004), which originates from the fact that both approaches use an effective flow angle and take similar liquid film thicknesses into account (see Figure 7 (a)). However, the values of this work consider a discretized packing sheet that includes areas with many different positive and negative effective liquid flow angles and the delft-model considers a single, global effective liquid flow angle value for the whole packing sheet. Furthermore, this work includes experimental measurements of film thicknesses on real plates with microstructures and the delft-model takes the theoretical solution of the Nusselt film thickness for smooth plates into account. Results of Billet&Schultes (1993) are between 31% to 41% higher than the values determined by the second experimental set-up (TUB) and constantly 49% higher than the solution of the delft-model. Billet&Schultes (1993) calculate the hold-up using the volume-specific area value of the packing as the only geometrical information, which is rather a rough estimation than a defined parameter.

The dynamic liquid hold-up curve of the first experimental set-up (RUB) in Figure 7 (b) is the average of all measurements in Figure 3 and compared to the semi-empirical correlation of Billet&Schultes (1999). Billet&Schultes (1999) include a packing-specific fitting constant of C_h that has been determined by various

hold-up measurements in lab-scale columns using the draining method as well. In this case, a C_h constant of 0.515 has been taken into account. The results of the first experimental set-up (RUB) and the correlation of Billet&Schultes (1999) identify good agreement especially for low liquid loads.
Both miniaturized experimental set-ups show comparable results to corresponding liquid hold-up correlations, which implies that experimental set-up 1 is able to predict the dynamic liquid hold-up and experimental set-up 2 is be able to predict the total liquid hold-up.

4. Conclusion and Outlook

An alternative approach to design structured packing columns more accurate and less time consuming is introduced, which includes a numerical cell-model and experimental investigations in a miniaturized set-up. Hence, two closely aligned experimental set-ups were developed and tested to find the minimum required size for the miniaturized experimental set-up that is able to achieve insight on fluid dynamics and mass transfer conditions of a real fluid/packing-system.
The measured liquid hold-up turned out to be independent from the number of packing sheets without a countercurrent gas flow but is actually depending on the microstructured surface of packing sheets. In fact, the MONTZ-Pak B1 microstructure increases the film thickness by 2% - 6% compared to the Nusselt solution for smooth surfaces.
Results for dynamic liquid hold-up measured with the draining method indicate good agreement to the semi-empirical correlation of Billet&Schultes (1999). Results for total liquid hold-up of the presented geometrical scale-up model using measured film thickness on microstructured surfaces slightly overestimate the holdup correlation of the delft-model (Olujic et al., 2004). Hence, both experimental set-ups intend to be able to determine either the dynamic or total liquid hold-up.
In the next step, the cell-model of Van Holt&Grünewald (2016) will be adapted to structured packing columns and connected to the results of the miniaturized experimental set-ups. The outcome of the cell-model will be further validated using liquid distribution tests in a laboratory scale column.
After the development and validation of the hydrodynamic part of the cell-model, the miniaturized experimental set-ups will be adapted to perform measurements of mass transfer coefficients with a water-based fluid system with low surface tension and viscosity, which will be further implemented into the design approach.

Acknowledgments

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