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Experimental and Theoretical Studies on Local Liquid Phase Distribution in Packed Columns

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Knowledge about liquid maldistributions in packed beds can be used to improve the scale-up and design of packed columns. The cell model presented in this work is especially designed to account for large scale maldistribution, in particular wall flow. The underlying idea is a representative cell volume, in which variables such as concentration and hold-up are calculated based on an ideally mixed cell. To describe wall flow, the behavior of wall cells differs from the bulk cells by means of two different liquid distribution parameters. An investigation of the model parameters demonstrates that wall flow quantity and development are described as suggested by literature and experiments. Moreover, the influence of the column diameter on wall flow is qualitatively correct. Model validation is performed by residence time distribution experiments across the cross section of the column using a wire-mesh sensor for different packed heights. In advance, the sensor is satisfactorily validated for the tracer experiments with reference to literature. Based on this, the distribution parameters of the model are fitted to experimental data.

1. Introduction

Liquid maldistribution is known to have a significant impact on the separation efficiency of mass transfer columns as the effective mass transfer area is reduced (Billet, 1995). Maldistribution itself can be divided into small scale and large scale maldistribution (Hoek, 1983). The former is caused by the packing itself and is characterized by local effects like channeling. The latter are maldistributions over the whole cross section of the column, e.g. wall flow or initial maldistributions (Hoek, 1983). While initial maldistribution can be reduced by an appropriate amount of distributor drip points, wall flow is caused by the anisotropy of the packing close to the column walls (Stikkelman, 1989). For industrial sized columns, the influence of wall flow on the mass transfer can be neglected (Grünewald et al., 2011). However, characterization of column internals is usually carried out in smaller columns and wall flow may be a major reason for the variations of mass transfer parameters measured at different test facilities (Schultes, 2013). These parameter uncertainties are resulting in large safety margins during scale-up (Schultes, 2013).

There are already various approaches to consider maldistributions by means of experimental methods as summarized by Hegely et al. (2017). However, the use of models allows a more detailed insight into the impact of maldistribution. Different depths of detail are known: Two-columns models, e.g. as suggested by Mullin (1957), which just take into account the impact on mass transfer. More complex CFD approaches for detailed flow simulation, but so far without consideration of the mass transfer (Soulaine et al., 2014). Dispersion based distribution models (Hoek, 1983) and so called cell models (Stikkelman, 1989) that assume representative cells with in itself uniform conditions.

The cell model introduced in this contribution aims to simplify the scale-up through an exact description of the dynamic and spatial wall flow phenomena. Validation is done using a wire-mesh sensor to characterize liquid distribution in the packing.

2. Model Development

2.1 Fundamental Ideas

The following points have been identified as advantageous for a more detailed modelling of the liquid phase distribution:

- The idea of distribution is based on a single random packing and is geometrically independent.
- Cells have a defined volume with specific variables such as concentration, hold-up and (wetted) surface and are ideally mixed.
- Cell behavior of the wall region differs from the one in the bulk, which is described by two distribution parameters, respectively.
- The gas phase distribution is ideal.

2.2 Geometry and Stream Directions

The geometry of the model is shown in Figure 1a. The resulting cell geometry is described in Figure 1b.



Figure 1: (a) Geometry of the overall model, (b) Single cell with directions and fluxes

The volume of an annular plane is divided into cells of different axial, radial and tangential orientation, which are equidistant. An exception are the cell segments close to the wall, whose radial width can be chosen freely. Since liquid flow back in the previous upper cell is not possible, there are a total of five flow directions for the liquid phase. This leads to the following mass and component balance equations Eq(1,2,3) of liquid flow for each cell:

$$\dot{V}_{\text{in,cell}} = \dot{V}_{\text{flux,down,in}} + \dot{V}_{\text{flux,r',in}} + \dot{V}_{\text{flux,t',in}} + \dot{V}_{\text{flux,t',in}}$$
(1)

$$\dot{V}_{\text{out,cell}} = \dot{V}_{\text{flux,down,out}} + \dot{V}_{\text{flux,r-,out}} + \dot{V}_{\text{flux,r+,out}} + \dot{V}_{\text{flux,t-,out}} + \dot{V}_{\text{flux,t+,out}}$$
(2)

$$\dot{V}_{\text{in,cell}} \cdot \mathbf{C}_{\text{in,cell}} = \dot{V}_{\text{flux,down,in}} \cdot \mathbf{C}_{\text{flux,down,in}} + \dot{V}_{\text{flux,f^{,in}}} \cdot \mathbf{C}_{\text{flux,r^{,in}}} + \dot{V}_{\text{flux,r^{+,in}}} + \dot{V}_{\text{flux,t^{+,in}}} \cdot \mathbf{C}_{\text{flux,t^{,in}}} + \dot{V}_{\text{flux,t^{+,in}}} \cdot \mathbf{C}_{\text{flux,t^{+,in}}}$$
(3)

The chosen geometry leads to changes in the area of the cell walls with each radial segment. Therefore, the definition of the distribution mechanism is crucial.

2.3 Distribution Mechanism and Cell Balance

The distribution of the liquid is mainly caused by the mass transfer equipment. Thus, the mechanism is designed to be based on a single random packing. Figure 2a illustrates the mechanism by means of the net liquid split streams for a single random packing. Figure 2b presents the link between the single packing and a representative cell that is based on the number of single packings at the walls of each cell.

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Figure 2: (a) Definition of the flows for a single packing, (b) Link to the distribution from a single packing to a cell

A single packing as well as the representative cell will distribute incoming liquid either through its four sides or downwards. The sum of the side streams and the downward stream is equivalent to the exiting flow. The ratio of the lateral to the downward streams is defined by the splitting factor k_{split}.

$$k_{\text{split}} = \frac{\dot{V}_{\text{side,packing}}}{\dot{V}_{\text{side,packing}} + \dot{V}_{\text{down,packing}}}$$
(4)

with

V_{out,packing}=V_{side,packing}+V_{down,packing}

(5)

(8)

Here k_{split} is an internal specific parameter that can be determined individually by experiments. The number of individual packings at the wall of the cell i is represented by Eq(6), assuming that the area covered by a single packing is a constant input parameter:

$$n_{\text{Packing,side i}} = \frac{A_{i}}{A_{\text{FK}}}$$
(6)

Therefore, the outflow from cell surface depends on the number of packings as shown in Eq(7,8,9):

$$V_{\text{side i,cell}} = n_{\text{packing,side i}} \cdot 0.25 \cdot V_{\text{side,packing}}$$
(7)

 $\dot{V}_{down,cell} = n_{packing,down} \cdot \dot{V}_{down,packing}$

$$\dot{V}_{\text{out,cell}} = n_{\text{packing,down}} \cdot \dot{V}_{\text{down,packing}} + \sum_{i=r^{+}rt^{+}t} n_{\text{packing,side }i^{+}} 0.25 \cdot \dot{V}_{\text{side }i,\text{packing}}$$
(9)

The factor 0.25 in Eq(7) and Eq(9) is caused by the four possible flow directions out of a single packing. Hence, k_{split} is the distribution influencing parameter between lateral and downward flow.

It is known that wall flow is a common maldistribution in mass transfer columns. Obviously, the flow behaviour on the wall differs from the one in the bulk. Therefore, a spatial differentiation of k_{split} in k_{bulk} and k_{wall} is necessary. Naturally, k_{wall} is lower than k_{bulk} , because of the lower porosity of the packed bed close to the wall. The balance equation for a cell of the model is assumed to be ideally mixed (CSTR):

$$\frac{dH_{L,cell}}{dt} = \frac{V_{in,cell} - V_{out,cell}}{V_{cell}} \text{ with } H_{L,cell \text{ at } t=0} = 0$$
(10)

$$\frac{dn_{i,cell}}{dt} = \dot{V}_{in,cell} \cdot \dot{C}_{i,in,cell} \cdot \dot{C}_{i,out,cell} \text{ with } c_{i,out,cell} = c_{i,cell}$$
(11)

To take into account a dynamic behaviour, the specific potential hold-up $H_{L,cell,pot}$ of each cell is calculated by the correlation of Billet and Schultes (1993). In steady state operation, the hold-up $H_{L,cell}$ of each cell is equivalent to the potential hold-up. Furthermore, it is assumed that the outlet flow velocity of a cell is quadratically dependent on the liquid hold-up, which is based on Torricelli's law (Anderssohn, 1870). Based on this, the connection between the inlet and outlet flow of each cell can be expressed by Eq(12):

$$\dot{V}_{out,cell} = \dot{V}_{in,cell} \cdot \sqrt{\frac{H_{L,cell}}{H_{L,cell,pot}}}$$
(12)

2.4 Model Verification

The main targets of the model are the prediction of wall flow development and quantity. Figure 3 illustrates these aspects for an ideal initial liquid distribution with variation of the splitting factors k_{bulk} and k_{wall} as well as the column diameter. Figure 3a shows no changes in the wall flow fraction with increasing packing depth with equal splitting factors, because the flow from the bulk to the wall and vice versa are equal. For k_{bulk} greater than k_{wall} , the radial outflow from the bulk region becomes larger than the incoming flow from the wall with otherwise constant model parameters. This results in lower cell hold-ups in the bulk region and thus lower downstream flows. In Figure 3b the influence of the column diameter is illustrated. As the diameter of the column increases, the wall flow fraction decreases, with the change in wall flow fraction not being proportional to the change in diameter. The modelled wall flow behaviour is qualitatively similar for to the one expected in real columns. This can also be shown for hold-up, influence of the gas load and liquid residence times.



Figure 3: (a) Wall flow development as a function of k_{bulk} and k_{wall} , (b) Wall flow development as a function of the column diameter

3. Model Validation

3.1 Experimental Setup

The wire-mesh sensor (Figure 4a) was introduced by Prasser et al. (1998) as a tomographic electrode mesh for measuring gas-liquid flows. It is composed of two wire planes, the transmitter and receiver plane, that are orthogonal to each other, building up a mesh like structure. The sensor can be operated in electrical permittivity or conductivity mode, while for both modes the calibration is performed with the pure components, e.g. water and air. As an output, the sensor directly measures the phase fraction between the planes. With 2,500 frames per second and a spatial resolution of 4.5 mm, as used in this contribution, the wire-mesh sensor is capable of resolving large and small scale maldistributions.



Figure 4: (a) Wire-mesh sensor with d = 288 mm and 64x64 mesh, (b) Conducted Tracer injections in the experimental setup

Experiments were carried out for the Pall Ring 25 in a column of 288 mm inner diameter and countercurrent gas liquid flow at packed heights of 0.5, 1.0 and 1.5 m. Liquid loads were measured with rotameters, gas loads according to DIN EN ISO 5167 based on the pressure drop along an aperture. Liquid was distributed

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using a pipe distributor with 553 drip points per m². The wire-mesh sensor was installed 17 cm below the packing. Uniform gas distribution was ensured by a packed height of 0.5 m Pall Ring 25 below the sensor. Distribution experiments were conducted with tracer pulses initiated in the center of the packing and close to the wall. Solutions of sodium chloride with concentrations adapted to the respective liquid loads were used as tracer. The residence time distributions were measured in the conductivity sensor mode. A schematic representation of the used experimental setup and procedure is given in Figure 4b.

3.2 Experimental Results

The capability of the wire-mesh sensor for the determination of tracer spreading was validated with reference to literature. Cihla and Schmidt (1958) introduced a dimensionless spreading coefficient, which is based on the radial propagation of the liquid over the packed height. These radial spreading coefficients were determined for three different heights using the wire-mesh sensor with a central tracer initiation. The influence of bed height is negligible, as shown by the standard deviation in Figure 5a. Without gas load, the results are in good agreement with data from Chromik (1992). The spreading coefficients determined by Stikkelman (1989) show a positive deviation from those of this work, which might be referred to differences in the column diameter, inlet conditions or the measurement techniques. Nevertheless, no reasons were found not to rely on the tracer detection capability of the wire-mesh. Therefore, the results were used for the determination of the splitting factor k_{bulk} . As the model is designed more phenomenological, with differentiated wall and bulk regions, the splitting factor k_{wall} was analogously determined by tracer distribution experiments on the wall.



Figure 5: (a) Comparison of the radial spreading coefficient by variation of the gas factor, (b) Fraction of detected tracer in the wall region (ring radius 135-144 mm)

Figure 5b shows the fraction of tracer detected in the wall region to the total tracer detected. At gas loads below the loading point, the fraction is approximately constant. The relatively large deviation is based on the measurement equipment. At gas loads above the loading point, the fraction of detected tracer in the wall region decreases. Thus, the gas flow in the wall region increases and displaces the tracer, due to the increased liquid flow from wall to bulk. Nevertheless, the amount of tracer detected in the wall region becomes very low at higher gas loads. Therefore, the pulse injection at a single point on the wall might be insufficient. Further investigations in the wall region will focus on a continuous tracer injection and an increased number of injection positions.

3.3 Model Validation

Since the simulation results meet expectations, the model was validated concerning the wall flow representation. The model geometry became independent from cell number for a cell height smaller than 50 mm and a radial width smaller than 8 mm. No influence of the tangential cell width was observed. Only the radial width of the wall region was fixed to 9 mm due to the validation with a 9 mm liquid collector. Using the experimental results shown in Figure 5a and 5b, both splitting factors were determined. Without countercurrent gas flow and for a column diameter of 288 mm, k_{bulk} and k_{wall} were determined to 0.2596 and 0.0790, respectively. The following Figure 6 shows the simulated wall flow in a column 500 mm diameter compared to the experimental results of Kouri and Sohlo (1996). The results obtained with the new model are in good agreement with literature. The slight deviations between simulation and literature can be explained due to different inlet distributions, the different randomly packed beds and the scattering of experimental data used for the determination of the model parameters in this work. Especially, the good determination of wall flow development is a major improvement of this model approach to previous models.



Figure 6: Comparison of the simulated wall flow to experimental results by Kouri and Sohlo (1996), $d=0.5 \text{ m} \mid u_L=18 \text{ m/s} \mid F_v=0 \text{ Pa}^{0.5} \mid \text{Pall Ring 25} \mid \text{Model parameters were fitted to experimental results at } d=288 \text{ mm}$

4. Conclusions

A new model approach to describe large scale distribution in packed columns was introduced. The differentiation in wall and bulk regions with specific behavior is the basis for the description of wall flow. Further innovations include the consideration of the defined cell volume, the cell hold-up and the cell concentration. These variables are important to describe the local distribution phenomena and the imminent implementation of mass transfer.

The formation of wall flow depends on the introduced splitting factors. In order to predict the wall flow, the model parameters were fitted to the spreading behavior in bulk and wall region. It has been shown that the model results are in good agreement with experimental wall flow data. The underlying measurement technique, the wire-mesh sensor, was validated with regard to tracer detection. Finally, mixing and distribution phenomena were found to be influenced by the gas load, since the liquid flow close to the wall decreased (for a Pall Ring 25) after passing the loading point.

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