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Thema: **Neues Simulationstool zum Scale-up auf Basis Populationsdynamik**

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INTRODUCTION

Particulate processes are of uttermost importance in chemical industry. Therefore, the mathematical modeling and simulation of e.g. droplet swarm behavior in columns are a necessary condition for scale-up, model-based feed-forward control and optimization. Because of their complex hydrodynamics the column behaviour is difficult to predict due to continuous change in the droplet properties such as Sauter mean droplet diameter, hold-up and concentration profiles for organic and aqueous phases. The state-of-the-art is the simple dispersion model, not taking into account the discrete nature of the dispersed phase. Alternatively, a drop population balance model (DPBM) is used. This takes into account the drop transport (rise and back-mixing) and drop interactions to the macro scale (drop breakage and coalescence) and at micro-scale (interphase mass transport). These droplet interactions are responsible for the evolution of the droplet size in multiphase flow. Different models in literature are studied and developed in the past decade to express these interactions but they are mostly dependent on column type, size and internal geometry besides the chemical test system. For these models most of them contain parameters that require fitting. So what is necessary is to have models and parameters that are independent on the column geometry which is essential for design of extraction column especially during scale up and scale down.

The main objective is to evaluate breakage and coalescence parameters. The validation is at steady and transient conditions, when using different operational step changes (rotational speed, flow rate) with different chemical test systems in a rotating extraction column (RDC, Kühni). In addition, to validating the optimized models with the online prediction and determination in extraction columns, using an in-house tool Online Monitoring and Simulation Tool (OMST) (Mickler et al., 2013). Also 3D-CFD parameter model validation by FPM, OpenFOAM and Fluent commercial software. Additionally a DFG-proposal was made, which is still in the review process.

RESULTS

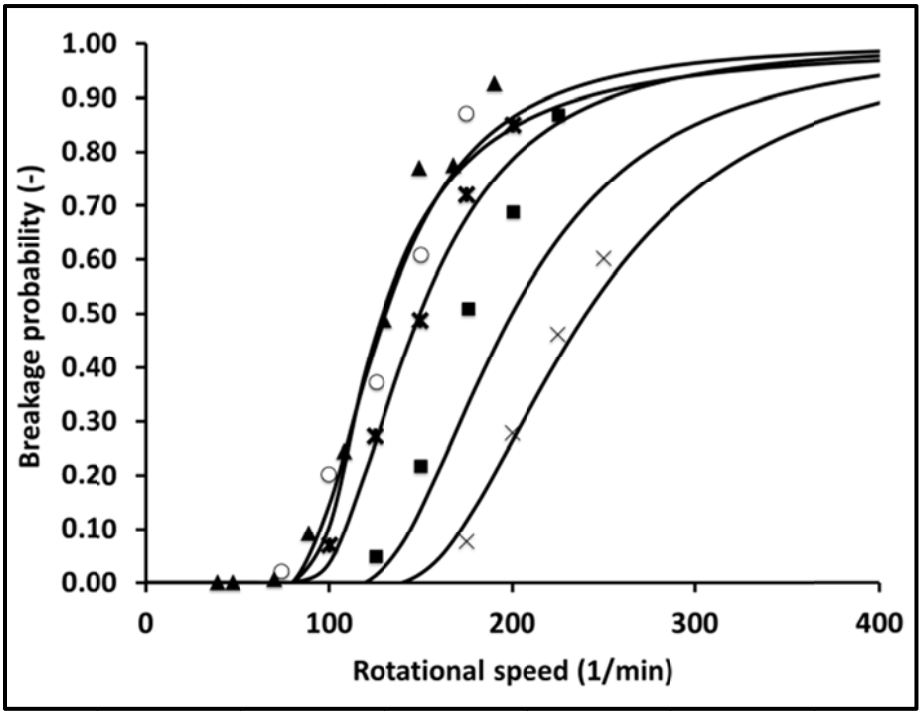
The parameter estimation problem is necessary to provide the droplet breakage and coalescence kernels or any other relevant parameters that could not be measured directly. Depending on experimental data availability, such as in industrial cases usually the only available data are the inlet and outlet conditions and few intermediate data along the equipment height. In such cases, the DPBM for both hydrodynamics and mass transfer has to be solved. Therefore, not only the size of the system is considerably increasing, but also the computational time due to the slow mass transfer process. For estimating the required parameters a solution of inverse DPBM problem is used, which is a well-known ill-conditioned problem and a careful algorithm design is required. Therefore, the parameter estimation is done by using the classes method (CM), and for an online process prediction and control the recent developed numerical model by Attarakih et al. (2009) so called One Primary One Secondary Particle Method (OPOSPM) is used and compared to the CM-PBM. The model consists of only two transport equations: namely, the continuity equation for the number and volume concentration, which is able to captures all the essential physical information.

For droplet breakage and coalescence model interactions parameters the optimization was done using MATLAB R2012b equipped with a global optimization toolbox package. The optimization algorithm is based on minimization of objective function using nonlinear least-squares data fitting with bounded parameter constrains to insure physical reliability of the estimated parameters.

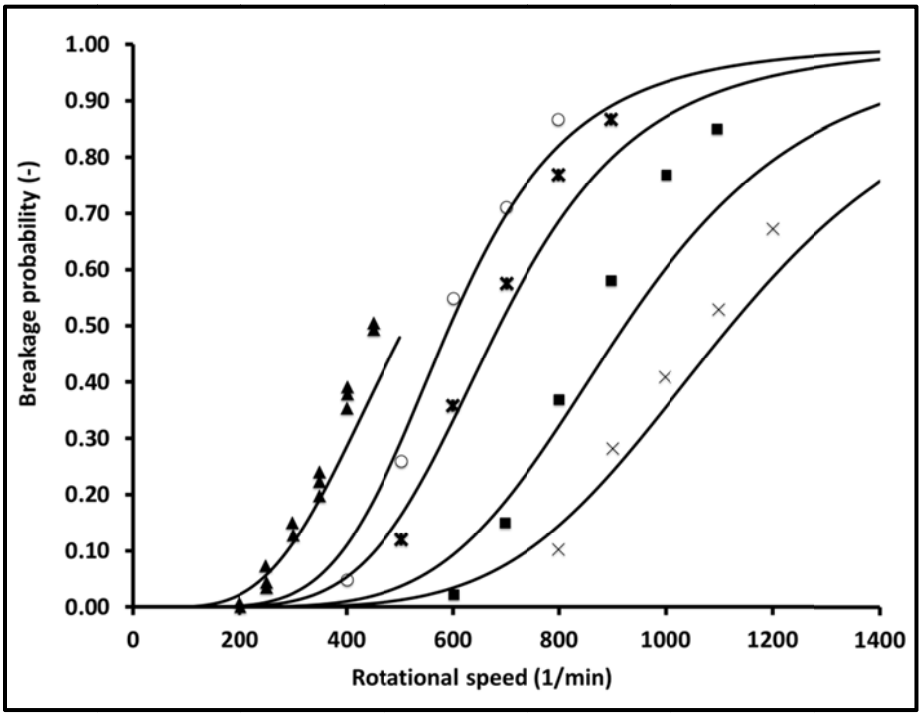
Droplet breakage is influenced by the hydrodynamics, droplet size, system physical properties and energy dissipation. It is described by various models in literature which contains parameters (Liao and Lucas, 2009). Different breakage probability models have been studied Schmidt et al. (2003), Schmidt (2006) and Garthe (2006). Breakage probability and quality of parameters are found strongly dependent on the magnitude of the experimental data used in parameter estimation. The single droplet experiments were collected from the work of Garthe (2006), Schmidt (2006) and Steinmetz (2007) for various binary and ternary test systems, rotational speed and mother droplet diameter in different Kühni and RDC compartment geometries.

As an example, the experimental data in Figure 1 are for a Kühni compartment (Figure 1a) and RDC compartment (Figure 1b) taken from Garthe (2006) with column diameter DN80 for four test systems: butylacetate (d)-water (ba-w), toluene (d)-water (t-w), butylacetate (d)-acetone-water (ba-a-w) and toluene (d)-acetone-water (t-a-w), and from Schmidt (2006) with column diameter DN150 for test systems isotridecanol (d)-water (iso-w) as recommended by the European Federation of Chemical Engineering (EFCE). The parameters have been optimized with the combined data set using MATLAB, where satisfactory results are shown in Figure 1 (lines).

A total of five correlation parameters (b_1 - b_5) in each model are dependent on compartment type but no longer on the chemical system. Further Kühni parameters validations for the influence of the interfacial tension, mother droplet diameter and scale down to DN32 miniplant column on the breakage probability is in Jildeh et al. (2013a).



(a)



(b)

Figure 1: Breakage probability for different test systems (× t-w, ■ t-a-w, ✱ ba-w, ○ ba-a-w and ▲ iso-w) a) Kühni compartment b) RDC compartment.

Droplet coalescence is considered to be more complex than breakage, because droplet interactions are not only from surrounding liquid phase but also by other droplets as they come into contact by external flow and body forces. Droplet coalescence is also highly sensitive to the hydrodynamics and physicochemical properties, interfacial dynamics and mass transfer. It is described by various models in literature which contains parameters (Liao and Lucas, 2010).

The inverse problem for droplet population balance models (DPBM) is solved to estimate the optimal coalescence of liquid extraction columns. Once these parameters have been optimized under certain conditions column, they can then be effectively used for predicting the behaviour of liquid extraction columns. The accurate simulation will lead to a replacement or at least minimizing the tests in pilot plants in extraction columns, resulting in reduced time and cost. The aim of this work is to find the best method and adjust the parameters not only to the experimental data, but also to consider whether the fitted parameters can predict the behaviour of the system under entirely different operating conditions. Five different models have been studied: Coulaloglou and Tavlarides (1977), Sovová (1981), Casamatta and Vogelpohl (1985), Laso (1986) and Lane et al. (2005) in Jildeh et al. (2012a and 2013b). Based on these previous studies done for RDC extraction column using different EFCE chemical system the model of Coulaloglou and Tavlarides (1977) is chosen to calculate the coalescence frequency see Figure 2. This model relies on physical properties, energy dissipation and holdup, droplet diameter and the two correlation parameters. Simulated Sauter droplet diameter and holdup of the disperse phase along the column height as compared to the experimental data for two test systems butylacetate (d)-acetone-water (ba-a-w) and toluene (d)-acetone-water (t-a-w) discussed more in details in Jildeh et al. (2012a and 2013b), further validation and concentration profile using LLECMOD is found in Jildeh et al. (2013b).

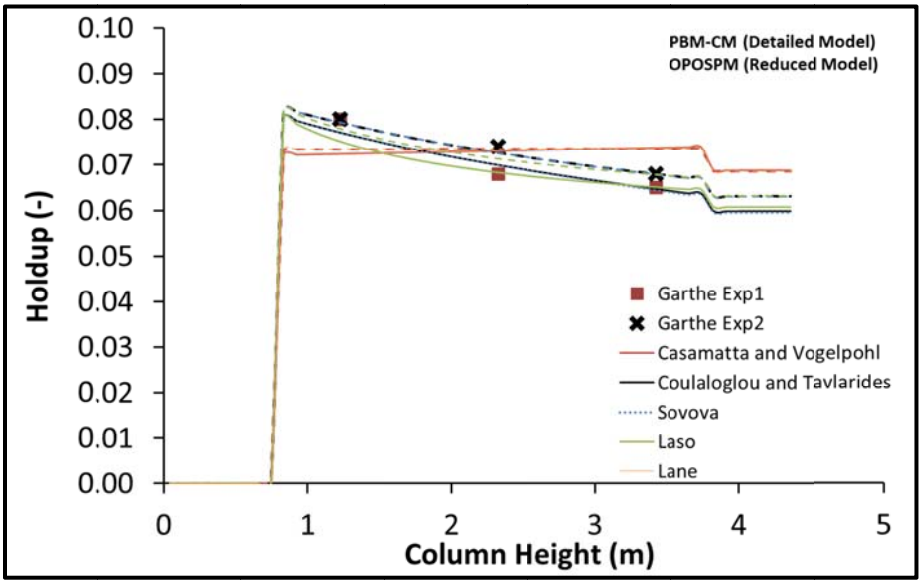
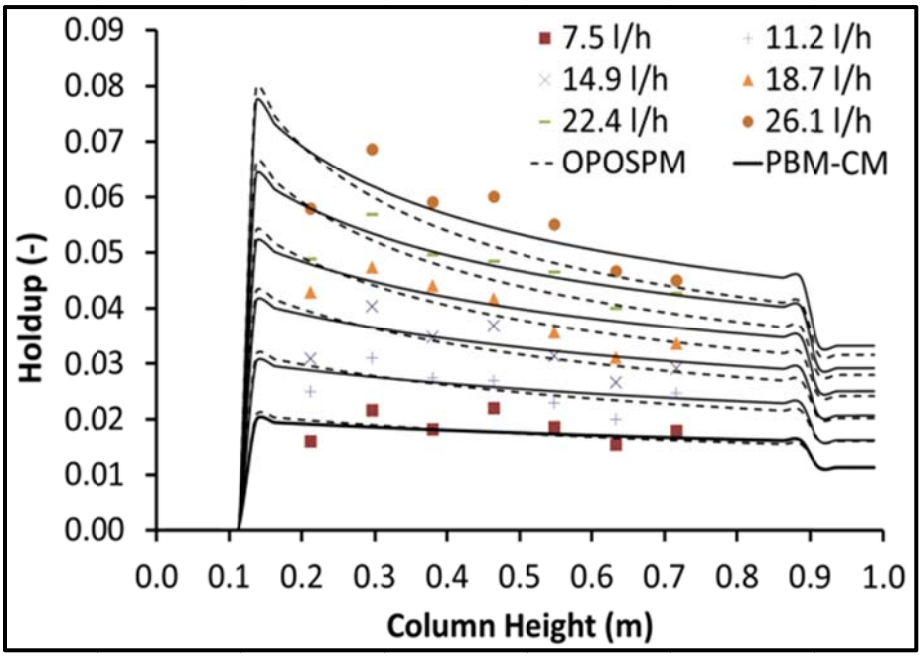
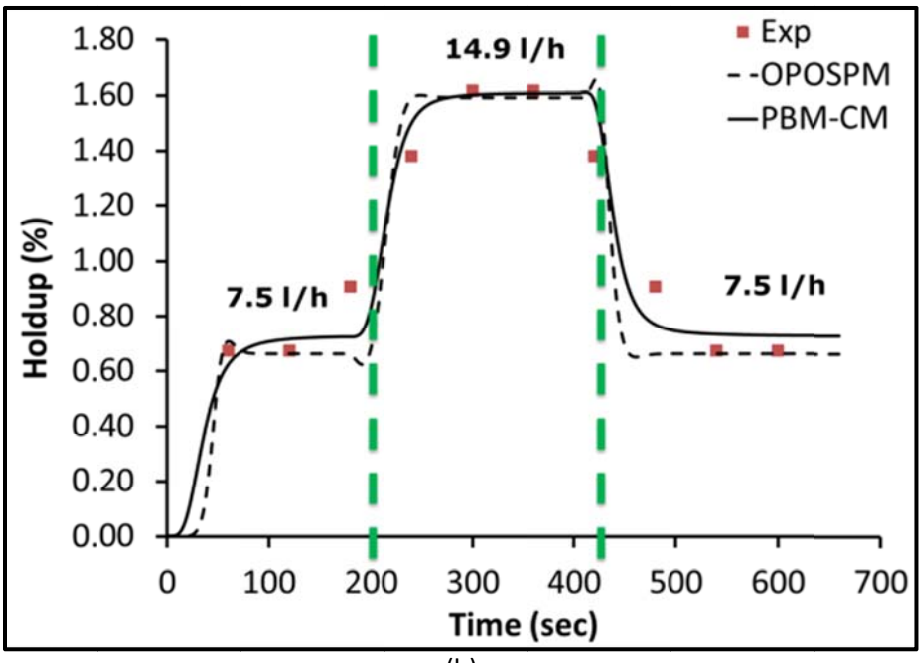


Figure 2: Simulations using the OPOSPM and PBM-CM compared to experimental data in RDC extraction column for toluene-acetone-water (Jildeh et al., 2012a).



(a)



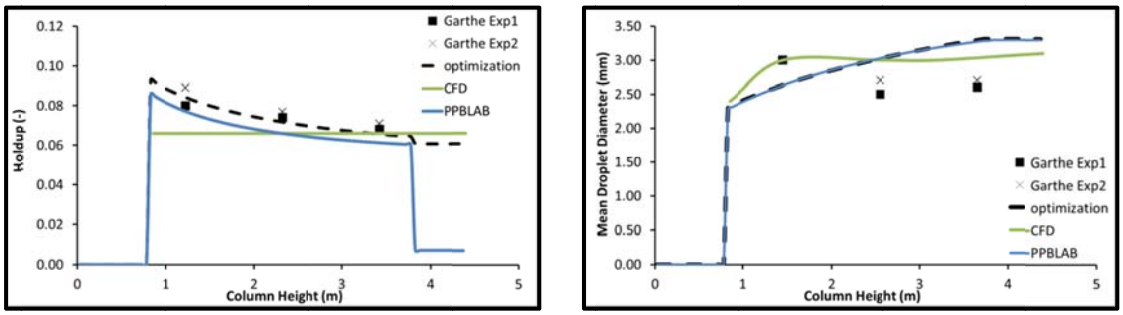
(b)

Figure 3: Simulations using the OPOSPM and PBM-CM compared to experimental data in Kühni extraction column (Jildeh et al., 2012b). a) Holdup at constant rotational speed 140 rpm b) Transient state with step change in flow rate 7.5-14.9-7.5 l/h.

Figure 3 is the steady and transient state simulation in Kühni extraction column DN150 for toluene-water. Theses offline simulation Figure 2 (RDC) and Figure 3 (Kühni) results show that the OPOSPM can easily simulate the liquid extraction column with less than 3% of the total simulation time required by CM-PBM.

A. CFD validation

The EFCE test system toluene-acetone-water (t-a-w) with weight fraction of acetone in the aqueous phase is equal to 0.05, is used for the CFD validation in a pilot RDC extraction column. The operating conditions used: rotor speed is constant at 400 rpm and the volumetric flow rate for the continuous and dispersed phase is 40 l/h and 48 l/h, respectively. A total of four parameters of droplet breakage and coalescence parameters for Coulaloglou and Tavlarides model (1977) are estimated by solving the inverse problem. Figure 4 shows the optimization results for solving the inverse population balance problem by using the experimental data for both the holdup and the droplet diameter along the column height as reference value. Using these optimized parameters, CFD simulations were performed to get local information about phase fraction, continuous phase velocity, energy dissipation and droplet size. The resulted holdup and droplet size of the optimization, the CFD simulation and PPBLAB with mass transfer is compared to the experimental results shown in Figure 4. Further CFD results and concentration of acetone in the dispersed phase and continuous phase compared to the experimental measurements is in Jildeh et al. (2012c).



(a) (b)

Figure 4: **Optimization of breakage and coalescence parameters using experimental data taken from Garthe (2006) for an RDC extraction for toluene-acetone-water test system used as input values for CFD and PPBLAB simulation (Jildeh et al., 2012b). a) Holdup b) Mean droplet diameter.**

B. OMST validation

The online prediction of transient process changes in complex multiphase flow is still challenging. The investigations have been carried for extraction column, to be able to do real-time predictions about future events. A comparison between the process information results (droplet size, holdup) collected from the experimental measurement techniques and robust simulation of system properties are necessary. For the simulation the improved OPOSPM with learning parameters was used based on an adapted moment's method for online simulation. As a result, of joint work M. Mickler-measurement techniques and H. Jildeh-modeling and simulation based on optimized models, the Online Monitoring and Simulation Tool (OMST) is programed and used to determine, analyze, simulate and predict the multiphase flow behaviour online in Kühni extraction column. For validation, steady state and transient experiments are performed, where rotor speed and dispersed volume flow rate are changed for toluene–water system. The OMST droplet sizes and holdup results from the

simulation and experimental results are in quite in agreement, further modifications are still under investigation. Figure 5 shows one validation which shows that the accuracy is highly depending on the quality of the parameter estimation. Sensitivity analyses for set of breakage and coalescence parameters are given in Jildeh et al. (2013a) for different test systems.

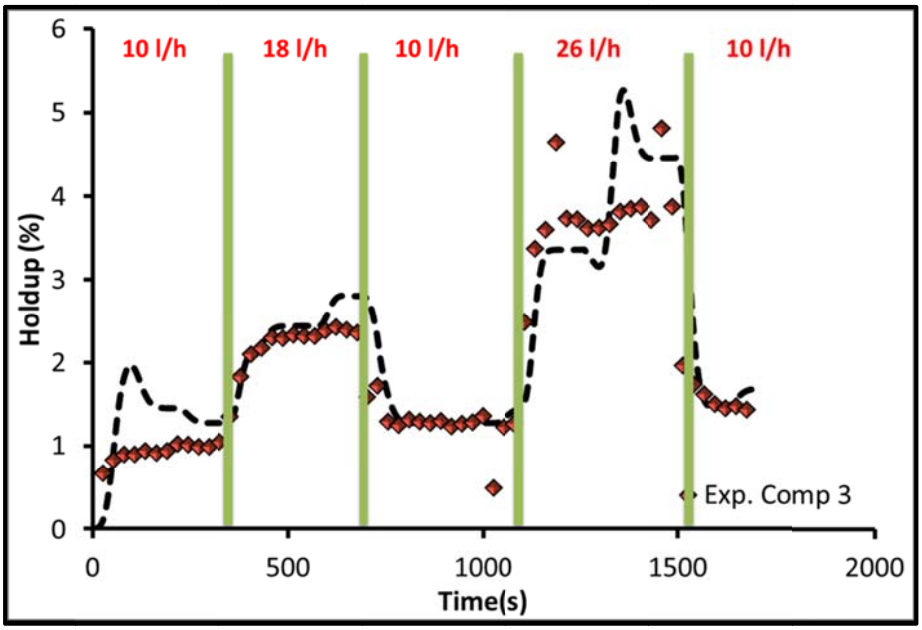


Figure 5: **Online transient validation for dispersed phase flow rate (10-18-10-26-10l/h) using the final set of breakage and coalescence parameters mentioned in Jildeh et al. (2013a)**

CONCLUSIONS

The inverse population balance was solved for the full CM-PBM to estimate the parameters required in droplet breakage and coalescence kernels, using different test systems in extraction column type and geometries. Different breakage and coalescence models were compared and optimized to estimate the model parameters. As could be shown, the parameter set is independent from deviations in column geometry and invariant to changing operating conditions. It is partly dependent on the chemical system used (only the coalescence), which gives rise to a further improvement of the coalescence models. These offline simulation results have been validated offline using 1D (LLECMOD, PPBLAB) and 3D CFD and online using the OMST. The results prove that the methodology to obtain insensitive parameter is accurate and which has a high advantage for designing and simulating extraction column especially during scale up and scale down.

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