Advanced Prediction of Pulsed Extraction Column Performance using LLECMOD

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Abstract— **A bivariate population balance model (the base of LLECMOD program) for the dynamic simulation of liquid extraction columns is extended to simulate pulsed and sieve extraction columns. The model is programmed using visual digital FORTRAN and then integrated into the *LLECMOD* program. In addition pulsed and sieve tray columns, *LLECMOD* simulates stirred two types of agitated columns (RDC & Kühni). As a case study, *LLECMOD* is used to simulate the steady state performance of a pulsed extraction column. Two chemical test systems recommended by the EFCE are used in the simulation. Model predictions are successfully validated against steady state and dynamic experimental data, where good agreements with the experimental data are achieved.**

I. INTRODUCTION

Liquid-liquid extraction is an important separation processes encountered in many chemical process industries [2]. Different types of liquid-liquid columns are in use nowadays, which can be classified into two main categories: agitated (RDC & Kühni) and pulsed (packed & sieve plate) columns. The latter are frequently used in liquid–liquid extraction operations due to their high throughput, high separation efficiency and insensitivity towards contamination of the interface. These columns have found wide applications in nuclear fuel reprocessing and chemical industry. They have a clear advantage over other mechanical contactors when processing corrosive or radioactive solutions. The absence of moving mechanical parts in such columns obviates the need for frequent repair and servicing. The internals (packing, sieve plates) reduces axial mixing; increases drop coalescence and breakage rates resulting in increased mass transfer rates, and affect the mean residence time of the dispersed phase. The performance of these columns is markedly dependent on the mechanical pulsation of the continuous phase. This is a result of an increase in shear forces and consequent reduction in size of dispersed droplets so that the interfacial area, and hence the mass transfer rate, is increased [3].

To shed more light on the extraction behaviour in pulsed (packed & sieve plate) columns, the hydrodynamics and mass transfer characteristics must be well understood. Our present knowledge of the design and performance of extraction columns is still far from satisfactory. The reason is mainly due to the complex interactions of the hydrodynamics and mass transfer [4]. It is obvious that the changes in the characteristics (holdup, Sauter diameter, etc.) of the drop population along the column have to be considered in order to describe conveniently the behaviour of the column. The dispersed phase in the case of liquid-liquid extraction undergoes changes and loses its identity continuously as the drops break and coalesce. Accordingly, detailed modelling on a discrete level is needed using the population balance equation as a mathematical framework. The multivariate non-equilibrium population balance models have emerged as an effective tool for the study of the complex coupled hydrodynamics and mass transfer in liquid-liquid extraction columns.

The simulation of modern industrial chemical processes is becoming extremely important as an economical tool in the integration of steady state and dynamic design as well as the simulation of the existing plants. The development of computational tools to model industrial processes has increased in the last decades. However; to the best of the authors' knowledge, there are no comprehensive non-equilibrium population balance models to describe in sufficient detail the behaviour of extraction columns. One of the recent approaches in modelling liquid-liquid extraction columns is by adequately describing the complex behaviour of the dispersed phase using the population balances equations (PBE). However, even the numerical solution of the resulting system of PBEs is still not efficiently developed particularly when coupled hydrodynamics and mass transfer take place simultaneously.

The main objective of this work is to develop a model that is capable of describing the dynamic and steady state behaviour of pulsed (packed & sieve plate) extraction columns. The models of both columns are integrated into the existing program: *LLECMOD* [1], which can also simulate agitated extraction columns (RDC & Kühni). *LLECMOD* can simulate the steady state and dynamic behaviour of extraction columns taking into account the effect of dispersed phase inlet (light or heavy phase is dispersed) and the direction of mass transfer (from continuous to dispersed phase and vice versa). So, scale-up and simulation of agitated and pulsed extraction columns based on population balance modelling can now be carried out successfully [1, 5].

II. MATHEMATICAL MODELLING

The modelling of extraction columns still demands improvement. Simulating liquid-liquid extraction columns is a

challenging task due to the discrete character of the dispersed phase. This results from random breakage and coalescence of droplets, which are highly coupled to the mass transport of solutes between the two existing phases. Modelling of such extremely important and complex transport phenomena is resolved by using a multivariate population balance equation. The population of droplets is modelled by a multivariate number concentration function, which takes into account droplet size and solute concentrations. Understanding of dynamic behaviour of liquid-liquid extraction columns can be notably used in the design of process control strategy or the start-up and shutdown procedures [6-9]. The attempts to model this dynamic behaviour are not well established because of the discrete nature of the dispersed phase. This is due to the complex nature of the macroscopic dispersed phase interactions as well as the microscopic interphase mass transfer occurring in the continuously turbulent flow field. These macroscopic interactions such as droplet breakage and coalescence coupled to the interphase mass transfer result in a distributed population of droplets. This population is distributed not only in the spatial domain of the contacting equipment, but also randomly distributed with respect to the droplet state (properties) such as size, concentration and age. Several attempts have been done to propose the proper mathematical model for liquid-liquid extraction columns [10]. An empirical model for predicting the hydrodynamics in a pulsed sieve plate column was proposed by Kumar and Hartland [11]. A stagewise model for the transient behaviour of a sieve-plate extraction column taking into account the back flow and assuming constant hold-up, was developed by Blass and Zimmerman [10]. Reference [6] evaluated a differential model in a Kühni column. Hufnagl et al. [12] modelled a packed column using differential contact model without axial mixing. Flow models such as the dispersion or backmixing model; describe the non-ideal flow, where one parameter accounts for all deviations from the ideal plug flow behaviour [13]. These models are too simple to describe the real hydrodynamics, where one of the liquid phases is normally dispersed as droplets in the second continuous phase [14]. Therefore, the influences of droplet movement, droplet interaction (breakage and coalescence), energy input (agitation, pulsation) and mass transfer cannot be described satisfactorily. Weinstein et al. [7] evaluated the differential model for a Kühni column. An improved dynamic combined model considering the influence of drop size distribution was developed by Xiaojin et al. [9]. Several population balance models have been proposed by various authors. Garg and Pratt [15] developed a population balance model for a pulsed sieve-plate extraction taking into account experimentally determined values for drop breakage and coalescence. Casamatta and Vogelpohl [16] proposed a population balance model for which a good review is found in [17]. Al Khani et al. [18] have applied this model for dynamic and steady-state simulations of a pulsed sieve-plate extraction column. Recently much works have been done in the population balance modelling for extraction columns [1, 17, 19-23].

A. multivariate non-equilibrium population balance model

The general spatially distributed population balance (SDPBE) for describing the coupled hydrodynamics and mass transfer in liquid extraction columns in a one spatial domain can be written as:

$$\frac{\partial f_{d,c_y}(\psi)}{\partial t} + \frac{\partial [u_y f_{d,c_y}(\psi)]}{\partial z} + \sum_{i=1}^{2} \frac{\partial [\dot{\zeta}_i f_{d,c_y}(\psi)]}{\partial \zeta_i} =$$

$$\frac{\partial}{\partial z}\left[D_y \frac{\partial f_{d,c_y}(\psi)}{\partial z} \right] + \frac{Q_y^{in}}{A_c} \frac{f_y^{in}}{\overline{v}_{in}}(d,c_y;t)\delta(z - z_y) + \Upsilon\{\psi\}$$

.. (1)

In this equation the components of the vector $\psi = [d\; c_y\; z\; t]$ are those for the droplet internal coordinates (diameter and solute concentration), the external coordinate (column height), z, and the time, t, where the velocity along the concentration coordinate (c_y) is $\dot{c}_y$. The source term $\Upsilon.\partial\zeta$ represents the net number of droplets produced by breakage and coalescence per unit volume and unit time in the coordinates range $[\zeta, \zeta+\partial\zeta]$. The left hand side is the continuity operator in both the external and internal coordinates, while the first part on the right hand side is the droplets axial dispersion characterized by the dispersion coefficient, D_y, which might be dependent on the energy dissipation and the droplet rising velocity [24]. The second term on the right hand side is the rate at which the droplets entering the LLEC with volumetric flow rate, $Q_{y,in}$, that is perpendicular to the column cross-sectional area, A_c, at a location z_y with an inlet number density, f_y^{in}, and is treated as a point source in space. The dispersed phase velocity, u_y, relative to the walls of the column is determined in terms of the relative (slip) velocity with respect to the continuous phase and the continuous phase velocity, u_x, with respect to the walls of the column [22, 23].

The solute concentration in the continuous phase, c_x, is predicted using a component solute balance on the continuous phase [22, 23]:

$$\frac{\partial(\phi_x c_x)}{\partial t} - \frac{\partial}{\partial z}\left(u_x \phi_x c_x + D_x \frac{\partial(\phi_x c_x)}{\partial z} \right) =$$

$$\frac{Q_x^{in} c_x^{in}}{A_c}\delta(z - z_y) - \int_0^\infty \int_0^{c_{y,max}} \dot{c}_y v(d) f_{d,c_y}(\psi)\partial d\partial c_y$$

(2)

Note that the volume fraction of the continuous phase, Φ_x, satisfies the physical constraint: $\Phi_x + \Phi_y = 1$, where y denotes the droplet phase. The left hand side of Eq.(2) as well as the first term on the right hand side have the same interpretations as those given in Eq.(1); however, with respect the continuous phase. The last term appearing in Eq.(2) is the total rate of solute transferred from the continuous to the dispersed phase, where the liquid droplets are treated as point sources. Note that Eq.(1) is coupled to the solute balance in the continuous phase given by Eq.(2) through the convective and the source terms.

B. Model parameters

The SDPBE is general for any type of extraction column. However, what makes the equation specific is the internal geometry of the column as reflected by the required correlations for hydrodynamics and mass transfer. Experimental correlations are used for the estimation of the turbulent energy dissipation and the slip velocities of the moving droplets along with interaction frequencies of breakage and coalescence [1, 22, 23].

C. Breakage probability and daughter droplet distribution

For pulsed packed extraction column the daughter droplet distribution is assumed to follow the beta distribution β, which is given by Bahmanyar and Slater [25]. In Eq.(3) below v is the average number of daughter drops per breakage event.

$$\nu = 2 + 0.34\left(\left(d'/d_{stab}\right) - 1\right)^{1.96} \tag{3}$$

$$\beta_n(d \mid d') = 3\,\nu\left(\nu - 1\right)\left(1 - \left(d / d'\right)^3\right)^{\nu-2}\left(d^5 / d'^6\right) \tag{4}$$

Here, d_{stab} is the stable droplet diameter, where droplets having diameter less than d_{stab} are not expected to break. The data for the stable drop diameters for pulsed packed and sieve trays columns under different operating conditions for the two standard EFCE test systems (water-acetone-toluene and water-acetone-butyl acetate) are given in [26]. The droplet breakage frequency and the daughter droplet distribution are correlated based on single droplet experiments. The droplet breakage frequency used in the simulation is given by [26]:

$$P_B\left(d\right) = C_1 \pi_{af}^{C_2} \frac{\left[\left(d - d_{stab}\right) / \left(d_{100} - d_{stab}\right)\right]^{C_3}}{C_4 + \left[\left(d - d_{stab}\right) / \left(d_{100} - d_{stab}\right)\right]^{C_3}} \tag{5}$$

Where π_{af} is a dimensionless number taking into account the influence of the pulsation intensity on the breakage probability and is given by [26]:

$$\pi_{af} = a.f.\left(\rho_c^2 / \mu_c.\Delta\rho.g\right)^{1/3} \tag{6}$$

This breakage frequency describes the breakage in a packed and sieve tray compartment with only one set of constant parameters for a liquid/liquid-system. The constants C_i appearing in Eq.(5) for pulsed (packed & sieve plate) columns are listed in [26]. To predict the breakage probability in pulsed columns with Eq.(5), the characteristic drop diameters d_{100} & d_{stab} have to be experimentally determined for each pulsation intensity. Data of the characteristic drop diameter for pulsed columns investigated here are listed in [26].

D. Droplet coalescence probability

In this work droplet coalescence probability for a pulsed extraction column is given by [27]:

$$P_C\left(d\right) = \frac{\varepsilon\, H_{cd}^{1/6}\,\left(\Delta\rho g\right)^{1/2}\Delta t\,\sigma^{1/3}}{\xi_8\,\mu_c\,d^{1/3}} \tag{7}$$

The parameter ξ_8 was fitted to experimental data for two standard EFCE test systems, for t-a-w ξ_8 is 2500, and for b-a-w ξ_8 is 1500 [27], Hamaker constant H_{cd} values used in the simulation are listed in table I & II.

E. The terminal droplet velocity

The terminal droplet velocity used in the simulation is given by [27]:

$$v_t = \frac{v_{os,de}v_{sph}}{\left(v_{os,de}^{a_{15}} + v_{sph}^{a_{16}}\right)^{1/a_{16}}} \tag{8}$$

In this velocity model, a_i the adjustable parameters can be fitted to the experimental data. v_{sph} is the spherical droplet velocity, $v_{os,de}$ is a smooth transition from oscillating to deformed droplets [27].

For the description of the drop motion in the structured packing, the correlation for the slowing factor developed by Garthe [26] is applied:

$$k_v = 0.077\pi_{H_{Pk}}^{0.138}\pi_{a_{Pk}}^{-0.566}\pi_d^{-0.769}\pi_\sigma^{0.184}\left(1 + \pi_{af}\right)^{0.08} \tag{9}$$

where π_{Hpk}, π_{apk}, π_d, π_σ, π_{af} are dimensionless numbers [26].

F. Mass –transfer

The mass transfer fluxes in the *LLECMOD* program are calculated based on the two-film theory, where the individual mass transfer coefficients are defined separately for the continuous (k_x) and the dispersed (k_y) phases. The mass transfer model used here in this simulation is taken from the work of Henschke [27].

G. Numerical solution

The mathematical model, which consists of integro-partial differential and algebraic equations is solved using an optimized and efficient numerical algorithms developed in [20-22]. These are utilized successfully for the simulation of coupled hydrodynamics and mass transfer for general liquid-liquid extraction columns.

III. LLECMOD PROGRAM

The complete mathematical model described above is programmed using Visual Digital FORTRAN. To facilitate the data input and output, a graphical user interface is designed. The graphical interface of the *LLECMOD* program contains the main input window and sub-windows for parameters and correlations input. The basic feature of this program [1] is to provide an easy tool for the simulation of coupled hydrodynamics and mass transfer in liquid-liquid extraction columns based on the population balance approach for both transient and steady states conditions through an interactive windows input dialog. Note that *LLECMOD* is not restricted to a certain type of liquid-liquid extraction column since it is built in the most general form that allows the user to input the various droplet interaction functions. These functions include droplet terminal velocity (taking into account the swarm effect) and the slowing factor due to column geometry, the breakage frequency and daughter droplet distribution, the coalescence frequency and the axial dispersion coefficients. Using *LLECMOD* simulations can now be carried out successfully for different types of extraction columns including agitated (RDC & Kühni) and pulsed (sieve plate & packed) columns. The design of *LLECMOD* is flexible in such a way that allows the user to

define droplet terminal velocity, energy dissipation, axial dispersion, breakage and coalescence frequencies and the other internal geometrical details of the column. The correlation parameters that are obtained based on single droplet and droplet swarm experiments, are considered in a modularized structure for the simulation program.

A. Coalescence parameter optimization package

As a new feature of *LLECMOD*, the program is reinforced by a parameter estimation package for the droplet coalescence models, which enables the user to fit the column hydrodynamics (droplet size distribution, holdup and mean droplet diameter) to the available experimental data. Prediction of the mass transfer profiles is then performed based on correlations obtained only from single droplet experiments. Simulation results are compared with data from pilot plant columns, such as agitated and pulsed/ un-pulsed columns. Different case studies showing columns' performance are presented using only optimized coalescence parameters. Additional results will be published in separate publications.

IV. RESULTS AND DISCUSSION

In this section a sample problem is considered to illustrate the basic features of the *LLECMOD* and the coalescence parameters estimation package. For this purpose, a pilot plant laboratory scale pulsed (packed & sieve plate) columns are considered whose dimensions are: column height $H = 4.4\ m$, inlet of the dispersed phase $z_y = 0.85\ m$, inlet of the continuous phase $z_x = 3.8\ m$, column diameter $d = 0.08\ m$. The two EFCE test systems toluene-acetone-water (t-a-w) and butyl acetate-acetone-water (b-a-w) are used whose physical properties are available online (http//dechema.de/extraktion). To completely specify the model, the inlet feed is fitted to a normal distribution with mean equals to *3.2 mm* and standard deviation of *0.5 mm*. The direction of mass transfer is from the continuous to the dispersed phase. All the operating conditions for the packed column are listed Table (I), and for sieve plate column in Table (II).

TABLE I
OPERATING CONDITIONS: PACKED COLUMN

Test System	Q_c lit/h	Q_d lit/h	$C_{y,in}$ %	$C_{x,in}$ %	af cm/sec	H_{cd} Nm
t-a-w	61.3	74.5	0.6	5.89	2	10e-10
b-a-w	62.0	72.0	0.0	5.21	2	10e-10

TABLE III
OPERATING CONDITIONS: SIEVE PLATE COLUMN

Test system	Q_c lit/h	Q_d lit/h	$C_{y,in}$ %	$C_{x,in}$ %	af cm/sec	H_{cd} Nm
b-a-w	62.0	72.0	0.0	5.46	1	10e-10
t-a-w	61.3	74.5	0.15	5.52	1	10e-12

Fig.(1) shows the variation of the mean droplet diameter along the height pulsed packed column and compared to the experimental data for both chemical systems. The agreement

between the simulation and experiment is excellent for both test systems.
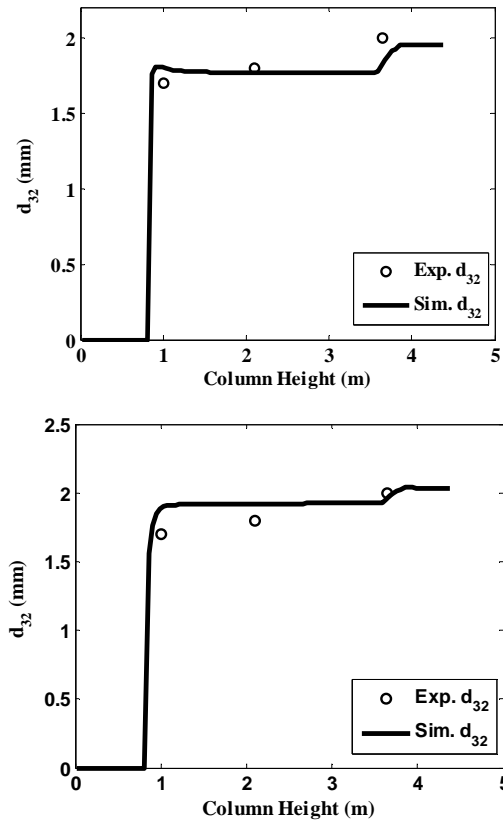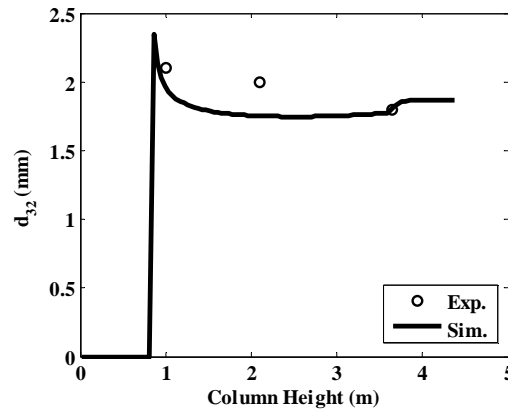

Fig. 1 Simulated mean droplet diameter along the column height compared to the experimental data [26]. Upper panel the test system is (b–a-w) and the lower panel is (t-a-w) in packed column.

Fig.(2) depicts the variation of the mean droplet diameter along the height of the pulsed sieve plate column and compared to the experimental data for both chemical systems. A fairly good agreement between the experimental and simulated profiles is achieved for both systems.
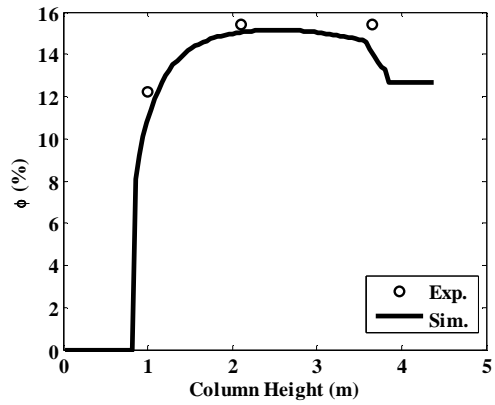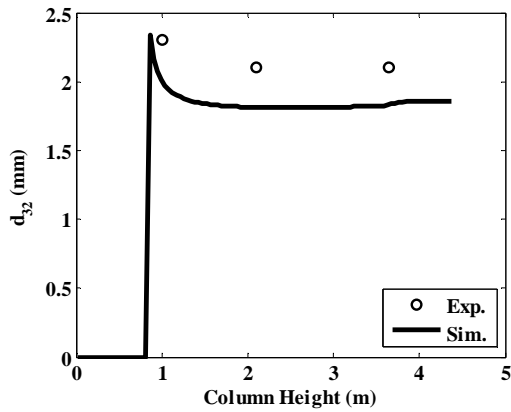
Fig. 2 Simulated mean droplet diameter along the column height compared to the experimental data [26]. Upper panel the test system is (b–a-w) and the lower panel is (t-a-w) in sieve plate column.

A comparison between the simulated holdup profiles along the height of the pulsed packed column and the experimental data [26] is shown in Fig.(3). Again, an excellent agreement is achieved for both test systems.
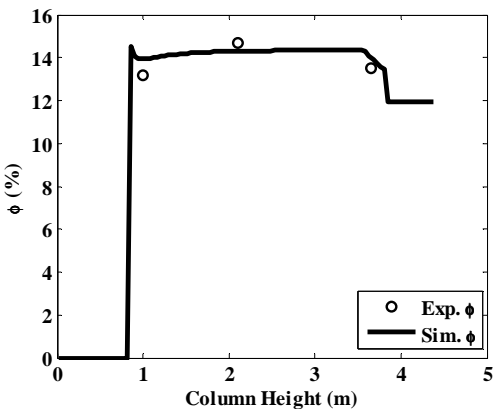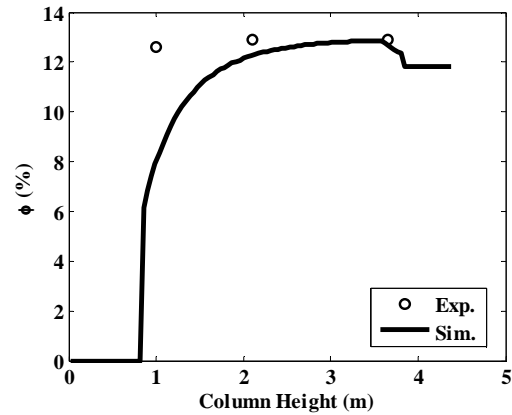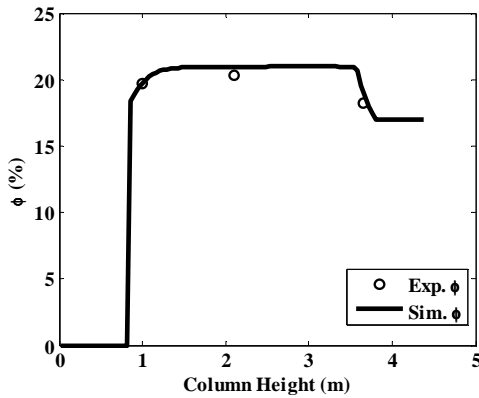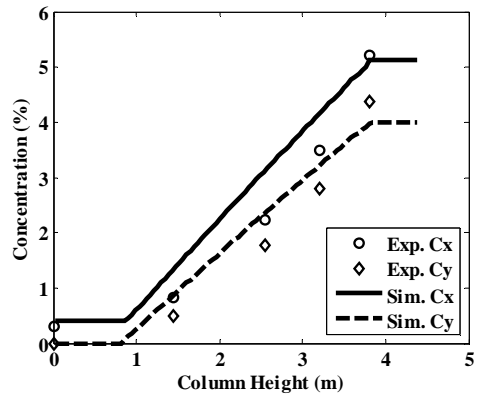

Fig. 3 Simulated holdup profiles along the column height compared to the experimental data [26]. Upper panel the test system is (t-a-w) and the lower panel is (t-a-w) in packed column.

Fig.(4) depicts the variation of the holdup profiles along the height of pulsed sieve plate column compared to the experimental data for both chemical systems. A fairly good agreement between the experimental and simulated profiles is achieved for both systems.

Fig. 4 Simulated holdup profiles along the column height compared to the experimental data [26]. Upper panel the test system is (b–a-w) and the lower panel is (t-a-w) in sieve plate column.

Fig.(5) shows the simulated and experimental solute concentration profiles as function of column height in both phases. The agreement between the simulation and experiment is very good for both test systems.

As a conclusion, the very good agreement between the simulated profiles and the experimental data appearing in Figs.(1-5) for different columns and chemical systems shows that LLECMOD capability of predicting the actual steady state performance of pulsed (packed and sieve plate) extraction columns.
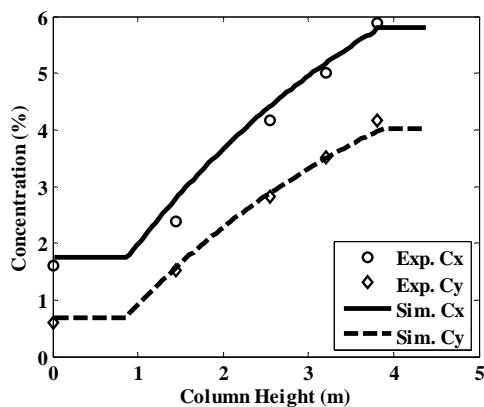
Fig. 5 Simulated solute concentration profiles in both phases along the column height compared to the experimental data [26]. Upper panel the test system is (b -a-w) and the lower panel is (t-a-w) in packed column.

Moreover, the dynamic behaviour of liquid extraction columns can be predicted using LLECMOD. This is necessary to improve the knowledge of the dynamic behaviour of extraction columns under the effect of different disturbances. Knowing how a system responds to disturbances is a prerequisite for controller design and optimization of column start up and shut down procedures. Further detailed research is underway to validate *LLECMOD* against available dynamic experimental data.

V. CONCLUSIONS

The LLECMOD non-equilibrium bivariate population balance model is found capable of simulating new types of extraction columns; namely, pulsed and un-pulsed packed and sieve tray columns in addition to the agitated columns (Kühni & RDC types). The rapid steady state hydrodynamics column behaviour can be efficiently predicted by only adjusting few parameters in the droplet coalescence model. These parameters can then be used to predict the very slow mass transfer process independently. In doing this, the model is validated successfully against the available experimental data.

ACKNOWLEDGMENT

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