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MODELLING AND IDENTIFICATION FROM BATCH EXPERIMENTS OF A SMB PROCESS

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Abstract: The Simulated Moving Bed (SMB) technology is a continuous chromatographic process which is important in various fields, from sugar to enantiomer separation. In this paper, a systematic identification procedure for determining parameters of SMB models from batch experiments is validated with experimental SMB data. Parameters are first estimated from elution peaks. Then a cross-validation with SMB experiments is performed so as to assess whether the parameters identified from batch experiments may be used in a SMB model. This part of the work requires a careful modelling of the dead volumes within the SMB process.

Keywords: identification, mathematical model, dead zones, validation, simulation

1. INTRODUCTION

The simulated moving bed (SMB) process is a continuous chromatographic separation process in which a counter-current movement of the liquid and solid phase is allowed by periodically switching the inlet and outlet ports. The transfer of the SMB technology, used industrially for hydrocarbon and sugar separation, to the separation of fine chemicals is not immediate. Indeed, the conditions and requirements (product quantities and purities, characteristics of the phases, interactions …) are very different. The main issues are the selection of optimal operating conditions and process control, problems which require the development of a model of the process. SMB models consist of mass balance equations in the liquid and in solid phases for the components to separate. These models usually contain a set of unknown parameters, which can be determined from batch experiments.

In the literature, many comparisons have been performed between experimental concentration profiles and simulated profiles. In most of the results presented, discrepancies are observed between the experimental profiles and the simulated ones. Two critical points are mentioned. On the one hand, the parameters are often roughly estimated from a few experiments (Strube et al., 98; Strube et al., 97; Païs et al., 97) or modified heuristically to minimize the difference between both profiles like in (Lehoucq *et al.*, 2000; Haag *et al.*, 2001). On the other hand, the dead volumes influence significantly the concentration profiles (Beste et al., 2000; Strube et al., 1998, Antos et al. 2001, Migliorini *et al.*, 1999).

The aim of this paper is twofold:

- to develop and validate a systematic procedure for estimating parameters of a SMB model from batch experiments;
- to propose an approach for modelling dead volumes.

The text is organised as follows. The presentation of the considered SMB process is given in Section 2. Section 3 is devoted to the parameter estimation from batch experiments. SMB modelling is discussed in Section 4 and cross-validation with SMB experiments is performed in Section 5.

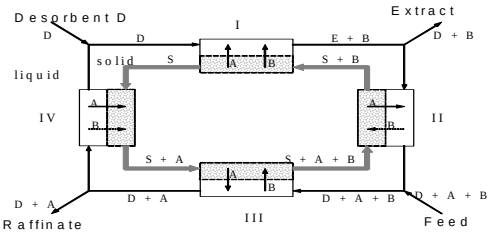

Fig. 1. Equivalent counter-current representation of a simulated moving bed process for separation of a mixture with two species A and B

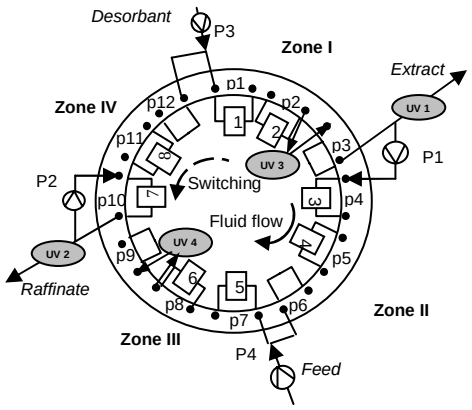

Fig. 2: Schematic representation of the SMB unit (MPI, Magdeburg, Germany) with 8 columns

2. SYSTEM DESCRIPTION

Fig. 1 shows the equivalent counter-current representation of a SMB process with the illustration of the movement of the liquid and solid phases, as well as the adsorption-desorption phenomena taking place in each section. The system is subdivided into 4 different sections delimited by several material flow outlets and inlets. The two inlets are the input of the mixture to be separated and the input of a desorbing solvent. The system also has two withdrawal ports, one for the raffinate which is mostly constituted of the less adsorbed component (component A) and another for the extract which consists of the most retained component (component B).

In this study, experiments were conducted at the Max-Planck-Institut Dynamik Komplexer Technisher Systeme in Magdeburg (Germany) on a preparative SMB unit (CSEP C912, Knauer, Berlin, Germany). Fig. 2 shows a schematic representation of this unit. In contrast with the usual configurations where the inlet and outlet ports are switched, the columns are connected to a multi-function valve and are switched in the counter-current direction to the fluid flow. This valve consists of a rotor and a stator with 24 ports each. The ports are connected to each other by continuous channels. Hence, all the devices inside the inner circle moves during the switching, whereas, the rest is fixed. Note that this SMB plant is built for up to 12 columns but only 8 columns are introduced in the process used in Magdeburg. Hence, as described in (Knauer, 2000), the free ports are connected by short capillaries and the valve switches alternatively one and two times successively during a full cycle (which is equal to 8 switching periods). The process is equipped with two inlet pumps, one on the feed flow (P4), and another on desorbent flow (P3). Two other pumps are located in the circulating stream (P1 and P2). Besides, this SMB process is also equipped with four UV detectors, two in the circulating stream (UV3 and UV4) and two on the product outlets (UV1 and UV2). In this study, it turns out that, in the considered concentration range, the UV signal is equal to a weighted sum of the concentration of both products.

The experiments consist to the separation of cyclopentanone and cyclohexanone on 8 columns with 21.2 mm internal diameter and 100 mm length. The stationary phase is a silica gel (LiChroprep RP – 18, particle size 25 – 40 µm, Merck, Darmstadt) and the solvent is a water/methanol solution (70/30).

The following conventions will be used in the paper:
- the columns are numbered (m = 1, 8); at the start-up, column 1 follows the desorbent input; column 2 is the following in the direction of the fluid flow, …
- a position (p) is defined as a place dedicated to the columns in the unit; some are really occupied by a column, others by a capillary replacing the column.

3. PARAMETER IDENTIFICATION FROM BATCH EXPERIMENTS

The method for determining the parameters of a SMB model from elution peaks is developed in this section.

3.1. Batch experiments

As it is assumed that all the columns of the SMB unit have the same properties, experiments will be performed on column 2 of the SMB plant which is followed by the detector UV3. By rotating the valve, it is placed after pump P3 (Fig. 2). A mixture with equal concentrations of component A (cyclopentanone) and B (cyclohexanone) is introduced at the top of the column thanks to a manual injection valve associated to pump P3. Elution peaks are measured at the bottom of the column with detector UV3.

3.2. Model and unknowns parameters

Column model and inlet concentration profile

The equations of the kinetic model for the m^{th} chromatographic column of the SMB unit are written as follows for the liquid phase:

$$\frac{\partial c_{i,m}}{\partial t} = -v_m \frac{\partial c_{i,m}}{\partial z_m} - \frac{1-\varepsilon}{\varepsilon}\frac{\partial q_{i,m}}{\partial t} \qquad (1)$$

with $c_{i,m}$, the fluid concentration, $q_{i,m}$, the solid

concentration, v_m, the fluid velocity, and ε, the porosity. t denotes the time and z_m, the axial coordinate. i = A, B refers to the species in the mixture to separate.
For the solid phase, the mass balance is given by:

$$\frac{\partial q_{i,m}}{\partial t} = k_{i,m}(q_{i,m}^{eq} - q_{i,m}) \cdot \quad (2)$$

with $k_{i,m}$, the mass transfer coefficient. $k_{i,m}$ is a linear function of the velocity in the column. $q_{i,m}^{eq}$, the adsorbed equilibrium concentration, is modelled by the Langmuir isotherm:

$$q_{i,m}^{eq} = \frac{q_{Si,m} b_{i,m} c_{i,m}}{1 + b_{1,m} c_{1,m} + b_{2,m} c_{2,m}} \quad (3)$$

with $H_i = q_{S,mi} b_{i,m}$. $q_{si,m}$ and $b_{i,m}$ are respectively the saturation capacity and the equilibrium constant of component i.

The inlet concentration profile is described as follows:
if $t < t_{Din}$ then $u_i(t) = 0$ (4)
else if $t < (t_p + t_{Din})$ then $u_i(t) = c_{i,F}\ (1 - \exp(-t/t_{tr}))$
else $u_i(t) = c_{i,F}(1 - \exp(-t/t_{tr})) - c_{i,F}(1 - \exp(-(t - t_p)/t_{tr}))$
with $c_{i,F}$, the injected concentration of component i, t_p, the injection duration and t_{tr}, a constant characterizing the rise time of the pulse (t_{tr} = 0.1 s). t_{Din} is the time delay due to the dead volume between the injection pump and the SMB process. It is implicitly assumed that the injection starts in t = 0 s.

Unknown parameters
The parameters that will be estimated are $\theta = [H_A \quad H_B \quad b_A \quad b_B \quad k_A \quad k_B]$.

Measurement equation
Letting $c_i(t, z; \theta, c_F)$, denote the solution of equations (1), (2), (3) for input (4) with c_F, a 2x1 vector with the injection concentrations of component A and B, the measurement equation can be written:
$y(t,\theta) = c_{A,m}(t, L; \theta, c_F)UV(A) + c_{B,m}(t; L, \theta, c_F)UV(B)$ (5)
where L is the column length. UV(A) (UV(B)) is the calibration coefficient of component A (B). It is determined by injecting successive step changes of known concentration of component A (B).

3.3. Statement of the identification problem

The unknown parameters are determined by minimizing a cost function which provides a measure of the deviation between the experimental profiles and the profiles simulated with the chromatographic model. The latter are obtained by solving numerically equations (1) – (5) following the method of lines (Schiesser, 1991; Haag *et al.*, 2001). To specify the parameter estimation problem, it is necessary to describe the parameter constraints and the cost function.

For each unknown parameter, prior knowledge allows one to specify an interval within which the estimated value must lie: $\theta(j)_{inf} < \theta(j) < \theta(j)_{sup}$. To enforce these constraints, the following non-linear transformation is performed on each parameter:

$$\theta(j) = 0.5(\theta(j)_{sup} + \theta(j)_{inf} + (\theta(j)_{sup} - \theta(j)_{inf})\tanh(\theta^*(j))) \quad (6)$$

with θ(j), the j^{th} parameter to identify, and $\theta^*(j) \in \Re$, the parameter which is actually determined by numerical optimization. Note that, for simplicity, by an abuse of notation, $y(t,\theta)$ is written $y(t,\theta^*)$ after parameter transformation.

Two data sets are used, one resulting from an injection at low concentration, S_1, the other from an injection at high concentration, S_2. Letting $y_{S_\ell}^{mes}(t)$, $\ell = 1,2$, denote the measured signal associated to the input concentration c_{i,S_ℓ}, the set S_ℓ can be defined by $S_\ell = \left\{ y_{S_\ell}^{mes}(t_{\ell,k}),\quad k = 0,1,...,M_\ell - 1,\quad t_{\ell,k} < t_{\ell,k+1} \right\}$
where injection is assumed to take place at time $t = t_{\ell,0}$.

The cost function is defined as:

$$J_{cl}(\theta) = \sum_{k=0}^{M_1-1} C_1 \left(y_{S_1}^{mes}(t_{1,k}) - y_{S_1}(t_{1,k}, \theta^*) \right)^2 + \sum_{k=0}^{M_2-1} \left(y_{S_2}^{mes}(t_{2,k}) - y_{S_2}(t_{2,k}, \theta^*) \right)^2 \quad (7)$$

with y_{S_ℓ}, $\ell = 1,2$, the simulated UV signals. C_1 is a constant that ensures that each data set has the same importance in the cost function:

$$C_1 = \max(y_{S_2}^{mes}) / \max(y_{S_1}^{mes}) \cdot \quad (8)$$

3.4. Solution of the parameter estimation problem

Optimization method
The optimization method used in this study is an algorithm for unconstrained optimization by quadratic approximation developed by Powell and called UOBYQA (Powell, 2000).

Multistart procedure
A multistart procedure is executed to alleviate the problem of local minima. It consists in performing 2^n identifications runs, with n, the number of parameters. Each run corresponds to a different value of the initial estimated parameter vector, $\theta^*_0{}^k$ (k = 1,.., 2^n). The latter are calculated after the following steps:

1. $\hat{\theta}_{init}$, a rough approximation of the parameters, is obtained from classical experimental methods applied directly on data sets S_1 and S_2.
2. $\Delta\hat{\theta}_{init}$, an upper bound of the error on $\hat{\theta}_{init}$, is estimated.
3. $\hat{\theta}^*_{init}$ and $\Delta\hat{\theta}_{init}{}^*$ are obtained after parameter

Table I: Batch identification conditions (column 2)

with $\varepsilon = 0.6$; $C_1 = 41.31$; $t_{Din} = 20$ s;

$v_m = 1.25^{e}$-5 m/s; $c_{S_1} = 0.12$ vol%; $c_{S_2} = 6$vol%

$\hat{\theta}_{init}$	[3 6.96 0.24 0.56 3.9 2.2]
$\Delta\hat{\theta}_{init} / \hat{\theta}_{init}$	[0.1 0.1 0.3 0.3 0.3 0.3]
θ_{inf}	[0 0 0 0 0 0]
θ_{sup}	[10 10 1 1 10 10]

Table II: Batch identification results

$J_{min}/(M_1+M_2) = 2.81e\text{-}5$

	$\hat{\theta}_{min}$	Confidence interval
H_A	3.05	[3.0429;3.057]
H_B	7.1	[7.08;7.12]
b_A (vol%)$^{-1}$	0.215	[0.2138;0.220]
b_B (vol%)$^{-1}$	0.66	[0.65;0.67]
k_A (s^{-1})	3.65	[3.5;3.8]
k_B (s^{-1})	2.38	[2.23;2.54]

transformation (6) of $\hat{\theta}_{init}$ and $\Delta\hat{\theta}_{init}$.

Finally, $\theta^{*}{}_0{}^{k}$, k = 1,..,2^n, corresponds to of the vertices of a hyper-parallelepiped centred around $\hat{\theta}^{*}{}_{init}$ with edge length equal to $2\,\Delta\hat{\theta}_{init}{}^{*}$. Each identification run yields an estimated parameter value denoted $\hat{\theta}*^{k}$, or after transformation by equation (6), $\hat{\theta}^{k}$, k = 1,.., 2^n. The associated value of the minimum cost function will be denoted $J_{min}{}^{k}$, k = 1,.., $2^{n.}$. Subsequently, J_{min} is calculated from $\min_k J^{k}_{min}$ and $\hat{\theta}_{min}$ is the parameter value for which J_{min} is reached. As it corresponds to the smallest cost function obtained, $\hat{\theta}_{min}$ is used further in validation tests.

3.5. Batch identification from experiments

Table I gives the identification conditions. The results of the identification and the confidence interval at 99% on the parameters, $\hat{\theta}_{min}$, calculated as described in (Seber and Wild, 1989) are given in Table II. As batch elution profiles are less sensitive to the mass transfer coefficients, the confidence interval is smaller for the isotherm parameters and larger for the mass transfer coefficients.

4. SMB MODELLING

In this section, the modelling of SMB processes is discussed in order to build the model that will be used to compare simulation results and SMB experiments to verify whether the parameters identified from batch experiments may be used in a SMB model.

4.1. Column Modelling

For column m of the SMB plant, equations (1) to (3) are valid. Note that $k_{i,m}$ is a function of the velocity in column m. This function is assumed to be linear. Hence, for each component, a relative mass transfer coefficient equal in all the columns of the SMB plant, is defined by $k_i^{rel} = \hat{k}_{i,m}{}^{batch} / v_{batch}$ (9)

with $\hat{k}_{i,m}{}^{batch}$, the mass transfer coefficient estimated from batch measurements and v_{batch}, the velocity used in the estimation step. Hence, $k_{i,m} = k_i^{rel} v_m$.

4.2. Switching

For simplicity, the fixed referential is associated to the columns. Hence, the switching is modelled by the movement of the inlet and outlet ports. To perform this, a vector which contains the flow rate at the twelve positions of the SMB plant is defined:

$$Q_p = [Q_{p1} \quad ... \quad Q_{p12}].$$

Valve switching is taken into account by considering that the flowrate at position p during a switching period is equal to the flowrate in position p-1 during the previous period:

$$Q_p{}^{n} = Q_{p-1}{}^{n-1} \qquad (10)$$

where n numbers the switching already performed. Hence, the velocity at position p is recalculated at each switching from $Q_{p.}$

4.3. Extra-column dead volume modelling

General Equation

The mass balance equation in the dead volume d is calculated as follows (Migliorini *et al.*, 1999):

$$\frac{\partial c_{i,d}}{\partial t} = -v_d \frac{\partial c_{i,d}}{\partial z_d} + D_d \frac{\partial^2 c_{i,d}}{\partial z_d{}^2}. \qquad (11)$$

with v_d, the velocity and D_d, the diffusion coefficient in the dead volume d. As most of the dead volume consists in tubes where plug flow conditions may be considered, D_d is small (≈1e-9 m²/s).

Switching of the dead volume in the circulating loop

In this experimental plant, a part of the dead volume is switched, like the detectors UV3 and UV4, and another part of the dead volume like the pumps is fixed. Hence, as seen in Fig. 3, at the n^{th} switching period, the dead volume at position p is divided into four parts, two moving, two fixed:

- the dead volume $V_{D,p}{}^{col,b}$ ($V_{D,p}{}^{col,af}$) which is located before (after) column m; it corresponds to the connection between the valve and the column m or connections between ports inside the multi-function valve; this dead volume switches with the columns at each switching time;
- the dead volume $V_{D,p}{}^{port,b}$ ($V_{D,p}{}^{port,af}$) which is situated before (after) column m; it corresponds for example to connections from (to) the pumps, P1 and P2, and connection from the inlet ports (to the outlet ports); this dead volume does not switch.

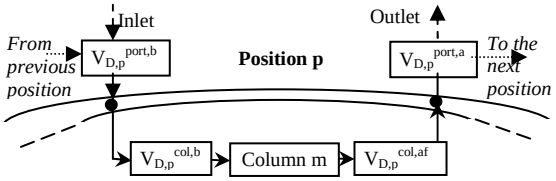

Fig. : 3 : schematic representation of the dead volumes at position p in the considered SMB unit

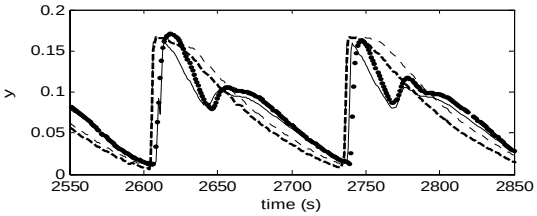

Fig. 4: Table IV: UV1 signal; • experimental signal; __ dead volumes in Table V; -- all the dead volume moving; --- $V_{D,p}^{port,b} = 0$ and $V_{D,p}^{port,af} = 0$

As the fixed referential is attached to the column, the switching of a part of the dead volume is modelled by considering that the concentration profiles in respectively dead volume, $V_{D,p}^{port,b}$ and $V_{D,p}^{port,af}$, at the beginning of a switching interval are equal to the profiles obtained in the dead volume $V_{D,p-1}^{port,b}$ and $V_{D,p-1}^{port,af}$ at position p-1 at the end of the previous period: (12)

$$c_{i,Dp}^{port}(t_s^n = 0, z_{Dp}) = c_{i,Dp-1}^{port}(t_s^{n-1} = \Delta t, z_{Dp-1})$$

with $c_{i,Dp}^{port}$, the concentration of component i in the dead volume $V_{D,p}^{port,b}$ or $V_{D,p}^{port,af}$ at position p. Δt is the switching period and t_s^n is the time elapsed since the n^{th} switching. z_{DP} is the position in the dead volume $V_{D,p}^{port,b}$ or $V_{D,p}^{port,af}$. Note that if position p is not occupied by a column, $V_{D,p}^{col,b}$ and $V_{D,p}^{col,af}$ are directly connected.

To illustrate the effects of equation (12) on the concentration profile, some simulations results are shown in Fig. 4. They allow to compare the experimental profiles recorded with detector UV1 and several results of simulation (for details about these simulations and measurements see Section 5). It appears that the only way to reproduce the shape of the experimental signal is to take into account that a part of the dead volume is moving and another is fixed during switching.

4.4. Inlet concentration profile

The start-up of the plant coincides with the beginning of the injection of a continuous feed flow in the process filled with solvent. Hence, the inlet concentration profile is described as follows:

$$\text{if} \quad t < t_{D_{in}} \quad u_i(t) = 0$$
$$\text{else} \quad u_i(t) = c_{i,F}(1 - \exp(-(t - t_{D_{in}})/t_{tr})) \qquad (13)$$

with $t_{D_{in}}$, the dead time introduced by the dead volume between the feed tank and the SMB unit. t_{tr} characterizes the rise time of the step (t_{tr} = 0.1 s). $c_{i,F}$ is the feed concentration of component i, i = A, B.

4.5. Measurement equations

The measurement equation is written:

$$y_{UVj}(t,\theta) = c_{A,D_j}(t, L_{D_j}; \theta, c_F)UV_j(A) + c_{B,Dj}(t, L_{Dj}; \theta, c_F)UV_j(B) \qquad (14)$$

with j, the sensor number, $c_{i,Dj}$, i = A, B, the concentration of component i in the dead volume before the UV detector j. L_{Dj} is the length of this dead volume.

4.6. Numerical solution of the model equations

Equations (1) - (5) with (9) to (14) are solved numerically following the method of lines (Schiesser *et al.*, 1991) with boundary and initial conditions described in (Haag *et al.*, 2001).

5. VALIDATION WITH SMB EXPERIMENTS

SMB experiments are performed on the unit described in section 2. The operating conditions are described in Table III and the UV calibration factors in table IV. Table V gives the dead volumes for each position at the start-up of the plant. Fig. 5 and 6 show the comparisons between the measurements, the signals simulated with the initial parameter roughly estimated, $\hat{\theta}_{init}$, and with the identified parameters $\hat{\theta}_{min}$ for the UV1 signal and the UV3 signal. The simulations with $\hat{\theta}_{min}$ gives a good approximation of the measurements. The differences between the experiments and the simulation results, which are very small, may be explained by errors in the calibration coefficients or small variations of parameters among the columns. The signals simulated with $\hat{\theta}_{min}$ are close to the ones obtained with $\hat{\theta}_{init}$. Indeed, the concentrations obtained at the outputs are not very high and the initial parameters corresponding to the linear part of the isotherm and describing the behaviour at low concentration, $\theta_{init}(1)$ and $\theta_{init}(2)$, are close to the identified ones. However, an improvement of the profile is shown at higher concentration in Fig. 6.

Table III: Operating conditions

Feed concentration (vol%) $c_{A,F} = c_{B,F}$	1.456
Switching time (s)	130
Flow rate in zone II (ml/min)	28.7
Flow rate in zone IV (ml/min)	27
Feed flow rate (ml/min)	8.4
Solvent flow rate (ml/min)	31.3

Table IV: Calibration factors of the UV detectors

detector	UV1	UV2	UV3	UV4
UV(A)	0.227	0.206	0.1991	0.215
UV(B)	0.219	0.199	0.1919	0.207

Table V : Fixed and moving dead volume

Position t = 0 s	$V_{D,p}^{col,b}$ (m^3)	$V_{D,p}^{col,af}$ (m^3)	$V_{D,p}^{port,b}$ (m^3)	$V_{D,p}^{port,af}$ (m^3)
1	1.88^{e}-6	1.07^{e}-6	0.65^{e}-6	0.92^{e}-6
2	1.88^{e}-6	3.07^{e}-6	0.19^{e}-6	0.92^{e}-6
3	0.95^{e}-6	0.14^{e}-6	0.19^{e}-6	2.23^{e}-6
4	1.88^{e}-6	1.07^{e}-6	5.3^{e}-6	0.92^{e}-6
5	1.88^{e}-6	1.07^{e}-6	0.19^{e}-6	0.92^{e}-6
6	0.95^{e}-6	0.14^{e}-6	0.19^{e}-6	0.60^{e}-6
7	1.88^{e}-6	1.07^{e}-6	0.65^{e}-6	0.92^{e}-6
8	1.88^{e}-6	3.07^{e}-6	0.19^{e}-6	0.92^{e}-6
9	0.95^{e}-6	0.14^{e}-6	0.19^{e}-6	2.69^{e}-6
10	1.88^{e}-6	1.07^{e}-6	5.59^{e}-6	0.92^{e}-6
11	1.88^{e}-6	1.07^{e}-6	0.19^{e}-6	0.92^{e}-6
12	0.95^{e}-6	0.14^{e}-6	0.19^{e}-6	0.60^{e}-6

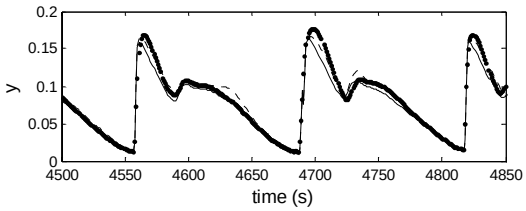

Fig. 5: UV1 signal; • experiments; -- simulation with the initial parameters; __ simulation with $\hat{\theta}_{min}$

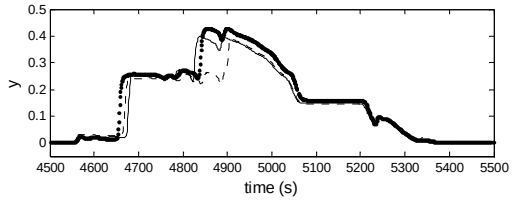

Fig. 6: UV3 signal; • experiments; -- simulation with the initial parameters; __ simulation with $\hat{\theta}_{min}$

6. CONCLUSIONS

In this work, a systematic procedure for estimating the isotherm parameters and the mass transfer coefficients of a SMB kinetic model is validated with experimental data. First of all, parameters are identified from two elution peaks, one at a small concentration, and another at a higher concentration. The confidence intervals calculated for each parameter are small. Then, these parameters are introduced in a SMB model so as to assess whether the parameters identified from batch experiments may be used in a SMB model. To this end, the introduction of the fixed and moving dead volumes in the model turns out to be necessary. The validation with SMB data is then performed with success.

7. ACKNOWLEDGEMENT

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