Optimal operating strategies for SMBC

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Abstract: Simulated Moving Bed Chromatography (SMBC) is a technical realization of the counter current adsorption process approximate by sequentially switching the inlet and outlet valves of interconnected columns in the direction of fluid flow. In this work, a systematic, simulation based optimization study is carried out for different operational goals in SMBC to enhance the performance of an existing laboratory setup. The optimization work involves Pareto optimal solution for two different conflicting objective functions. Optimal transition between such operating conditions is a challenging task. The quantitative results obtained by comparing the optimal transition method with a non-optimal, step change method shows that the optimal transition requires less time to achieve the new reference cyclic steady state. All of the above methods are studied on a SMBC process for separation of glucose and fructose using Ca^{++} exchange resin.

Keywords: Simulated moving bed chromatography, optimal operating transitions, dynamic optimization, separations process, glucose-fructose system

1. PROCESS DESCRIPTION

Chromatography is a separation technique based on the different adsorption affinities of the components of a mixture. In comparison to other thermal separation methods like distillation, less energy is consumed. Simulated moving bed chromatography (SMBC) process is a technical realization of counter-current chromatographic process known as true moving bed (TMB). SMBC has emerged as a new powerful separation technique for high value products in the pharmaceutical and fine chemicals industry (Rekoske, 2001). The main disadvantages of SMB are the dilute product streams that may need further concentrating and the cyclic nature of the process, which makes its operation difficult. The latter problem has been addressed in literature through optimization, wherein the internal flowrates and switch times are adjusted in order to extremize multiobjective performance metrics like purity and throughput. These multi-objectives are usually conflicting and the optimal operating strategy must represent an acceptable trade-off. For example, maximizing only throughput will result in maximum processing of feed with no or very little separation, that is, low purity. Moreover, due to the continuous-discrete nature of the process, transiting from one operating point to another is a non-trivial task. The intermediate product obtained during the transit between the two cyclic steady state operating points may not meet the spec and represents an economic loss. These optimal recipes would then serve as targets to a supervisory level control such as MPC. Therefore, there is a need to implement optimal transitions. In the context of start-ups and shutdowns, Li et al., 2011 has demonstrated the need for

optimal recipes so that the cyclic steady state (CSS) is rapidly achieved.

In the present work, we study optimal transitions between optimal operating points. Our results indicate that using optimal transitions help the SMBC operation reach the new setpoint more rapidly than if the operating parameters corresponding to the new setpoint were implemented in a step-like fashion. The paper is organized as follows: Section 2 presents a brief overview of the SMBC process and a summary of a first principles model from the literature. Section 3 discusses the optimal cyclic steady state operations. Optimal transitions between these operations are presented in Section 4. Finally, conclusions are presented in Section 5.

2. SIMULATED MOVING BED CHROMATOGRAPHY

The process consists of multiple columns which are connected in series in a circular manner as shown in Figure 1. In between columns, there are incoming fluid streams (feed and desorbent) or outgoing streams (extract and raffinate). The feed and desorbent are supplied continuously and simultaneously extract and raffinate are drawn through the ports. A counter current movement of solid stream is approximated by sequentially switching the inlet and outlet ports of interconnected columns in the direction of fluid flow. According to the position of columns connected to the feed and desorbent nodes, the SMB is divided into four sections each with a specific function in separating the feed mixture (see Fig. 1). The internal flow rates are different in each section. Consider the separation of a feed stream consisting of component A (more adsorbed) and B (less adsorbed). By choosing an appropriate switching time interval and flow rates, the preferentially adsorbed species A can be withdrawn at the extract outlet and the less adsorbed species B appears at the raffinate outlet. As

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Fig. 1. Schematic of 4-column SMBC unit

seen from Fig. 1, the feed alongwith the internal flow from section 2 enters section 3, where component A is preferenitally adsorbed on the stationary phase, while component B is carried by the liquid phase up to the raffinate outlet. The eluent is used to regenerate the adsorbent by desorbing component A in in the A-rich section 1 and component B adsorbs in section 4. The SMB unit reaches CSS after several cycles. At CSS, the transient concentration profiles of both liquid and solid at the end of the switch period are identical to the beginning of the period of the downstream adjacent column.

In this article, glucose-fructose separation is considered as a test bed to study optimal operations of SMBC corresponds to experimental setup of 1-1-1-1 column configuration as shown in Figure 1. Separation takes place on a strongly acid cationic resin of gel type (Ca^{2+} form) on which fructose gets preferentially adsorbed relative to glucose. Deionized water is used as the desorbent. So, fructose is drawn out in extract, which is considered as the main product.

2.1 Mathematical model of SMBC

In order to determine optimal operation of the SMB, a rigorous dynamic model of the continuous chromatographic process from literature is used, which can describe both interconnected columns and port switching operations. In this study, a rate-dispersive model (Rajendran et al., 2009) is used to obtain the concentration profiles of glucose and fructose in the columns. The model uses linear, noninteracting adsorption isotherms for the two species. A linear driving force model is assumed for the adsorption of the two species from the bulk phase to the stationary phase. We summarize the model equations here but cite the above reference for details (Rajendran et al., 2009; Kawajiri and Biegler, 2006).

Mass balance for i^{th} species in fluid phase:

$$\frac{\partial c_{ij}}{\partial t} + \frac{(1-\epsilon)}{\epsilon} \frac{\partial q_{ij}}{\partial t} = -v_j \frac{\partial c_{ij}}{\partial z} + D_i \frac{\partial^2 c_{ij}}{\partial z^2} \qquad (1)$$

where c_{ij} , q_{ij} represent the conentration of the i^{th} component in the j^{th} column in the liquid and solid phases, respectively and v_j represents the axial velocity in the j^{th} column. Axial dispersion and mass transfer effects are lumped into dispersion coefficient D_i .

Mass balance for i^{th} species in solid phase: A linear driving force is assumed to determine the solid phase concentrations,

$$\frac{\partial q_{ij}}{\partial t} = k_i \left(q_{ij}^* - q_{ij} \right) \tag{2}$$

 $i = 1, \dots, n_c$, the number of components $j = 1, \dots, NC$, the number of columns

where q_{ij}^* is the equilibrium concentration of i^{th} component in the j^{th} column in the solid phase. It has been widely reported that for dilute solutions, the glucose and fructose adsorption is non-competitive and follows linear adsorption isotherm on Ca^{2+} ion exchange resin,

Initial condition

$$c_{ij}(z,t=0) = c0_{ij}(z)$$
 (4)

 $q_{ij}^* = K_i c_{ij}$

The Danckwerts boundary condition at the column inlet yield (Leo and Rodrigues, 2004),

$$c_{ij}|_{z=0^{-}} = c_{ij}|_{z=0^{+}} - \frac{D_i}{v_j L} \frac{\partial c_{ij}}{\partial z}|_{z=0^{+}}$$
(5)

$$\left. \frac{\partial c_{ij}}{\partial z} \right|_{z=L} = 0 \tag{6}$$

(3)

Mass balance on the section nodes with respect to port positions shown in Fig. 1 yields,

Desorbent inlet node

$$Q_1 = Q_4 + Q_D \tag{7}$$

$$c_{i1}^{in}Q_1 = c_{i4}^{out}Q_4 \tag{8}$$

Extract outlet node

$$Q_2 = Q_1 - Q_E \tag{9}$$
$$c_{22}^{in} = c_{23}^{out} = c_E^E \tag{10}$$

Feed inlet node

$$Q_3 = Q_2 + Q_F \tag{11}$$

$$c_{i3}^{in}Q_3 = c_{i2}^{out}Q_2 + c_i^F Q_F$$
 (12)

Raffinate outlet node

$$Q_4 = Q_3 - Q_R \tag{13}$$

$$c_{i4}^{in} = c_{i3}^{out} = c_i^R \tag{14}$$

All the nodes are shifted after each switch period to the next position along the liquid flow direction. This creates a simulated counter current movement of solid phase. At the beginning of each switch, initial and boundary conditions for each column are updated according to concentration profiles in the column at the end of previous switch. The above model is converted to dimensionless form by introducing spatial and time dimensionless variables and are used in the optimization case studies presented later (see Rajendran et al. 2009 for details).

Model parameters corresponding to the SMBC and the glucose/ fructose separation are summarized in Table 1. These parameters correspond to the experimental setup at Automation Lab of IIT Bombay.

3. OPTIMAL OPERATION OF SMBC

Optimal operation of SMBC processes aims to determine the optimal operating parameters which satisfy various goals. Some goals are summarized below, • Maximize throughput, that is, feed flow rate: Often, we may desire to process maximum feed rate. Thus, referring to Figure 1, the throughput may be defined as

$$Q_F = Q_3 - Q_2 \tag{15}$$

• Maximize average extract purity: High purity is generally required in drug manufacture. Maximum purity of fructose is key in the manufacturing of corn syrup. The sweetness of corn syrup increases by increasing the amount of fructose which is more sweeter than glucose (Klatt et al., 2002).Let us assume that product identified as component A is preferentially adsorbed and is drawn from the extract stream (in our case, the product is fructose).Then, in the maximal extract purity mode of SMBC operation, the purity of the extract stream average over a switch time t^* at cyclic steady state is maximized. Thus,

$$Pur_{Ex} = \frac{\int_{0}^{t^{*}} c_{Fru,E}(t) dt}{\int_{0}^{t^{*}} c_{Glu,E}(t) dt + \int_{0}^{t^{*}} c_{Fru,E}(t) dt}$$
(16)

• Maximize extract recovery: In some situation, we may not desire pure product but that an expensive component (say, fructose) in the feed be maximumly recovered, thus

$$Rec_{Ex} = \frac{Q_E \int_{0}^{t^*} c_{Fru,Ex}(t) dt}{Q_F (\int_{0}^{t^*} c_{Fru,F}(t) dt + \int_{0}^{t^*} c_{Glu,F}(t) dt)}$$
(17)

As discussed previously, these multiple objectives represent conflicting operating strategies. Thus, any operating strategy based on optimization of one of the above objectives must ensure that the other objectives are maintained at acceptable levels.

3.1 The optimization strategy

The main objective of using optimization is to determine optimal operating parameters for optimal CSS operation that maximize some objective. Kawajiri and Biegler (2006) and Toumi et al. (2007) suggested two approaches for the cyclic steady state optimization problem namely, the simultaneous and sequential approach. In this work, we adopt the simultaneous approach, wherein the model equations form part of the equality constraints of the optimization problem.

Since we are ultimately interested in optimal transitions, we formulated the optimization problem by discretizing the model partial differential equations (PDE) Eqs. (1)-(14) over n switches, which is chosen large enough so as

Table 1. Parameter for Glucose/Fructose Separation

Parameter	Values	Parameter	Values
$d_c \ (cm)$	2.54	k (s^{-1})	0.1
L_c (cm)	40	ϵ	0.4
K_{fru}	0.5634	NC	4
K_{glu}	0.3401	C_{alu}^F, C_{fru}^F	30
-		(g/l)	

 Table 2. Initial operating conditions for Glucose/Fructose Separation

Variable	Values	Variable	Values
$Q_1 \ (\text{ml/min})$	42.23	$Q_3 (\text{ml/min})$	39.56
$Q_2 \ (\text{ml/min})$	36.56	$Q_4 \ (\text{ml/min})$	28.89
$Q_F ({ m ml/min})$	3.00	t^* (sec)	218.80

to achieve a cyclic steady state of the process. Kawajiri and Biegler (2006) formulated an optimization problem based on this method using Eq. (15) as objective function over a single shift and used the CSS condition as an additional constraint. Advantage of such an approach is that it eliminates time consuming CSS convergence during optimization. The method of orthogonal collocation on finite element was proven to be very efficient for solving fixed bed chromatography problem (Toumi et al., 2007). We used the roots of the shifted Legendre polynomial for discretization on each spatial as well as the temporal finite elements of equal lengths. An open source software IPOPT (Interior Point Optimizer) has been used in this work for solving the optimization case studies in which the equations used were coded in Matlab (R2007a). The fully discretized model Eqs. (1)-(14) are used as equality constraints. The spatial domain is discretized with 20 number of finite element per column and 2 internal collocation point per finite element. This was found to be appropriate by comparing with several discretizations schemes. The time domain is discretized by 4 internal collocation points per switch time. Initial conditions for operating variable such as internal flow rate and switch time are shown in Table 2.

3.2 Optimization case studies

In this work we study two different operation goals: namely maximize throughput Eq. (15) and maximize extract purity Eq. (16). The degrees of freedom used are the three internal flowrates namely, Q_2 , Q_3 , Q_4 , and the switch time (t^*) . The value of Q_1 is fixed at equal to 42.23 ml/min. All other variables used in SMBC are fixed upon deciding the above degrees of freedom. The various equality constraints consist of the mass balances Eqs. (1)-(6) and nodel balances Eqs. (7)-(14). The inequality constraints include non-negative values of the three external flow rates and the switch time. The total number of decision variables are 72964 which is calculated as [(Spatial finite element per column (6) x collocation point per element (2) + spatial finite element +1) x (No. of columns (4)) x (No. of phases (2)) x (No.of component (2)) x (temporal collocation point (4)]. Thus, the optimization problem consists of 72960 equality constraints and four lower bound constraints on the external flow rates and the switch time. Fig. 2 shows the time-varying concentration profiles of fructose and glucose in the extract and raffinte when the unit is operating in CSS.

3.3 Optimization Case 1: Maximize throughput in presence of recovery and purity constraints on fructose

There exist fundamental tradeoffs that make optimization of SMB multiobjective in nature. To account for this, Subramani et al. (2003) carried out study of multiple



Fig. 2. CSS Fructose concentration in extract

objective optimization in SMB with different combination of objective functions. They converted the multiple objective optimization problem into a single objective optimization problem by giving different weights to each of the objective function. In this study, we use the ϵ constraint method (Deb, 2001) to solve the multi-objective optimization problem. This method uses only one of the multi-objectives in the objective function and treats the remaining objectives as constraints whose lower bound is ϵ . The resultant problem is solved repeatedly by changing the value of the lower bound of ϵ . In this case study, the objective is to maximize throughput, that is, the feed flow rate of SMB, while the purity and recovery of fructose are treated as additional constraints. Note that the recovery is defiend as the fraction of the fructose that is recovered in the extract. This problem can be formulated as

$$\max_{c_{ij},q_{ij},Q_l,t^*} Q_F \tag{18}$$

s.t.
$$Rec_{Ex} \ge Rec_{,Ex,min}$$
 (19)

t.
$$Pur_{Ex} \ge Pur_{Ex,min}$$
 (20)
Eqs (1) - (14)
 $Q_l, t^* > 0$ $l = 2, \cdots, 4$
 $Q_1 = 43.23$ ml/min

We fixed $Rec_{,Ex,min}$ at 80% and the optimization problem was solved for different values of $tPur_{,Ex,min}$ ranging from 90% to 96%, .

s.

Results of the optimization runs for various values of are summarized in Table 3. Since the two objectives of throughput and purity represent a trade-off, we get a nondominating set of optimal solutions for different values of constraints on purity and are called Pareto set. It can be seen that the switch period changes significantly to increase product purity. It is clear that a large switching period improves the solid regeneration in section 1 of SMBC

Table 3. Case 1: Optimal results for Multiobjective optimization runs

	0.0,000	iio opt		on run			operation of SMB is a difficult task. Moreover, if manufac
Parameter							turers would like to integrate the operation with marke
$Q_F \ (\text{ml/min})$	2.92	2.90	2.87	2.791	2.712	1.939	1.069 conditions, it is imperative that the operational strateg
$Q_E \ (\text{ml/min})$	8.50	8.57	8.67	8.78	8.9	9.05	9.11 should respond quickly to account for any fluctuations
t^* (sec)	228.26	229.83	231.88	234.2	238.07	241.26	246.07 which is typical in large scale production of xylene in th
$Pur,_{Ex}$	90	91	92	93	94	95	<u>96</u> petrochemical industry. Results indicate that the optima
Avg. fru conc	8.24	8.61	7.94	7.62	7.16	5.15	2.81 operating strategy varies for different operating strategies
Rec, Ex	80	80	80	80	80	80	$\frac{80}{1000}$ Since the same unit is used to produce compounds wit
Avg. glu conc	8.82	8.83	8.80	8.78	8.16	6.24	$\frac{3.60}{3.60}$ different range of specifications on purity throughput etc
CPU (min)	5.03	5.16	5.94	5.51	9.61	16.7	$\frac{12.54}{12.54}$ the transition from one operating strategy to the other
IPOPT iter	6	7	7	8	13	23	21 must be performed entimely so that a quick charge y
							must be performed optimally, so that a quick changeove

and fructose adsorption in section 3. Note that thethe extract purity and recovery are at their lower bounds indicating the multi-objective nature of the solution. Table 3 also notes the CPU time taken to solve optimization problem on an Intel Core2Duo CPU with 2 GB RAM.

3.4 Optimization Case 2: Maximum Extract Purity

s.

The Maximization of extract purity problem can be formulated as

$$\max_{c_{ij},q_{ij},Q_l,t^*} Pur_{Ex} \tag{21}$$

t.
$$Rec_{Ex} \ge Rec_{Ex,min}$$
 (22)

(23)

$$Q_F \ge Q_{F,min}$$

Equation (1) - (14)
$$Q_l, t^* > 0 \quad l = 2, \cdots, 4$$
$$Q_1 = 43.23 \text{ ml/min}$$

As in case 1, the inequality constraints in equation (22)-(23) are imposed to get an optimal solution with a minimum acceptable value of feed flow rate and fructose recovery. The minimum value of recovery and feed flow rate at CSS 80% and 1 ml/min. IPOPT gives an optimal solution

Table 4. Results for Case 2

Parameter	Max fructose
	purity
	(Case 2))
$Q_F \ (ml/min)$	1.39
$Q_2 \ (\text{ml/min})$	34.08
$Q_3 \ (\text{ml/min})$	35.47
$Q_4 \ (\text{ml/min})$	28.89
t^* (sec)	245.9
$Q_D \ (\text{ml/min})$	14.80
Purity of fructose	96.25
Recovery of fructose	80
Avg fru conc	3.639
Avg glu conc	5.523
Purity of glucose	92.42
Recovery of glucose	93.8
CPU time (min)	11.18
Number of iterations	18

in 11.18 min. Optimization results are shown in Table 4. Results show that high purity is obtained only as dilute product at the expense of fed rate.

The next section discusses the optimal transition between two optimal CSS operating points.

4. OPTIMAL TRANSITION BETWEEN OPERATING STRATEGIES: CASE STUDIES

Because of continuous discrete nature of SMBC, optimal

is effected to reduce off-spec product. Hence, an optimal scheme has to be implemented in which process can move from one operating condition to desired operating condition in an optimal manner.

This issue is also encountered during startup of the SMBC. (Li et al., 2011) shows that the conventional start up procedure of SMB, where operating conditions corresponding to the cyclic steady state(CSS) operation are injected, take considerable time for the CSS to be reached. Hence, dynamic changes in operating conditions need to be injected to steer the plant to its CSS quickly. (Abunasser and Wankat, 2004) and (Rodrigues et al., 2007) provide startup procedure for single column chromatography analogue to SMB. Recently, (Li et al., 2011) give new multistage optimal startup and shutdown strategies in which operating parameters change transiently. Prata et al. (2008) studied such optimization cases in continuous polymerization reactor. To the best of the author's knowledge, in case of SMB, optimal transitions between optimal operating points have not been reported in literature. The main aim of optimal transition is to drive a process from an initial condition towards its reference condition in an optimal manner. While this problem becomes a dynamic optimization problem, we use control vector parameterization to convert it to a nonlinear program, which can then be solved using IPOPT. Since we do not know the time horizon necessary to obtain this transition, we parameterized the decision variables over a horizon of 28 switches, which was obtained by simulation trials. The amplitude of every decision variable is held constant during a switch period but allowed to change between switches. Therefore, the total number of degrees of freedom increases to 4*28 = 112. The objective function of the optimal transition problem is formulated as a multi-objective criterion, which represents a weighted sum of the deviations from the target CSS operating conditions, as follows:

$$\min \quad \phi = \omega^T X \tag{24}$$

where, ω is weighted vector documented in Table 5 and X are different objective function vector

$$X = \begin{bmatrix} \sum_{k=1}^{28} (Pur_{Ex,k} - Pur^{*})^{2} \\ \sum_{k=1}^{28} (c_{fr,k} - c_{fr}^{*})^{2} \\ \sum_{k=1}^{28} (c_{gl,k} - c_{gl}^{*})^{2} \\ \sum_{k=1}^{28} (Q_{T,k} - Q_{T}^{*})^{2} \\ \sum_{k=1}^{28} (t_{k}^{*} - t_{ref}^{*})^{2} \end{bmatrix}$$

$$\omega^{T} = [\omega_{Pur} \ \omega_{c_{fr}} \ \omega_{c_{gl}} \ \omega_{Q_{T}} \ \omega_{t^{*}}]$$
(25)

where n represent an upper bound on the number of switches required to attain the CSS. The objective function (24) is solved with the mass balance (1)-(14) and inequality constraints corresponding to the bound on the 128 dof. Several optimization trial runs were made to find appropriate value of weighting factors. The total time required to attain new optimal condition is calculated as,

$$t_{conv} = \sum_{k=1}^{28} t_k^* \tag{27}$$

where t^* represent switch time for the k^{th} shift. The optimal transition between the maximum throuhput (Case 1) operation to maximum purity (Case 2) is discussed in next subsection.

4.1 Case 3: Optimal transition from maximum throughput to maximum extract purity condition

It is assumed that the system is initially operating at CSS with optimal parameters corresponding to the maximum throughput as shown in Table 3. The new optimal case is maximum purity condition shown in Table 4. The objective function (24) is solved with degrees of freedom equal to 112. The solution was found in 63.46 min on a Intel Core2Duo machine with 2 GB RAM. The optimal transition operation takes only 12 switches to attain the reference profile. The actual transition period is equal to 49.26 min calculated using Eq. (27). The results are summarized in Table 6.



Fig. 3. Case 3: Evolution of fructose purity and concentration for optimal transition

Table 5. Case 3: Optimization result of optimal transition problem

Parame	ter Values	Parameter		Values
ω_{Pur}	0.2	Objective	function	35.07
		value		
$\omega_{c_{fr}}$	0.45	CPU time (r	min)	63.46
$\omega_{c_{al}}$	0.2	Number of it	terations	146
$\omega_{Q_T}^{g_*}$	0.05	Decision Var	riable	112
ω_{t^*}	0.1			

Fig. 3 shows the variation of the switch time-averaged fructose purity and concentration during the transition process. Variations in optimal operating conditions are plotted in Figs. 4 and 5. It is seen that in initial stages Q_2 is large and at the same time Q_3 reaches a low value, which corresponds to the lower bound in Q_F of 0.001 ml/min indicating that the feed entering into the column is negligible. Also, the switch time increases to ensure high desorption of fructose in column 1 which helps in increasing fructose purity. The raffinate flow increases in the very first step to discharge out more quantity of glucose so that it cannot pollute the extract in later switches. An opposite scenario happens with extract flow rate whose value decreases to increase the internal flow in section 2. All these changes occur in the initial switches.



Fig. 4. Internal flow rate profile for optimal transition method



Fig. 5. External flow rate profile for optimal transition method

Case 3A: A non-optimal transition using a step change in operating conditions To compare the benefits of optimal transitions, a non-optimal transition was simulated by using the operating conditions (that is, Q_2, Q_3, Q_4 and t^* with initial conditions corresponding to the maximum throughput case) corresponding to the maximum extract purity and then letting the system evolve to the new CSS. Figure 3 shows evolution of fructose switch-time averaged purity and concentration as a function of number of switches. This illustrates that SMB reaches its reference purity in 180.32 min or 44 switches to reach the new cyclic steady state calculated using equation (27), which is more than three times that needed in optimal transition. These comparisons are shown in Table 6.

5. CONCLUSIONS

The cyclic nature of SMB technology makes operation of SMB difficult. In this work, we studied the optimal operation of SMBC for separation of a glucose-fructose

Table	6.	Compa	rison	of	diffe	erent	transition
	stra	ategies	(Case	3 a	and (Case 3	3A)

Deremeter	One step	Ontimal
Farameter	One-step	optillar
		transition
Total transition period (h)	3.083	0.819
Feed supplied (lit)	0.249	0.0347
Desorbent consume (lit)	2.67	0.8111
Number of shift	44	12

system. It was shown that transitions during the operation can lead to lapse of significant time before the unit can be steered to the new CSS. In particular, it was shown that use of optimal transitions provides significant advantages over a step-change in the control variables of the plant.

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