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Two-Section Simulated Moving-Bed Process

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ABSTRACT

The continuous separation of glucose and fructose aqueous mixtures was experimentally performed by using a two-section simulated moving bed (SMB) process with Dowex 50W-X12 resin of Ca^{2+} form as an adsorbent and water as an eluant. The operation mode of the two-section SMB was modified from that of a three-section SMB developed by Barker et al. (1). A plug flow model with velocity-dependent mass transfer resistance was used for estimating the concentration profiles of the two-section SMB process, and the model equation was solved by the numerical method of orthogonal collocation. The two-section SMB suggested in this work was successful for obtaining high fructose corn syrup (55–90% w/w fructose).

INTRODUCTION

The development of countercurrent chromatography has been carried out along three different routes over the last 40 years, depending on whether the bed or the column has been physically moved (moving-bed or moving-column chromatography) or the stationary-phase movement simulated by mechanical means (simulated moving-bed process). A typical apparatus for moving-bed chromatography was used by Barker et al. (2) for a gas chromatography application to separate the azeotropic mixture benzene (bp 80.1°C) and cyclohexane (bp 80.7°C). Barker et al. (3) later modified his moving-bed apparatus to a moving column system for use with ternary hydrocarbon mixtures by introduction of a side arm containing a fresh flowing stream of packing between the bottom stripper and the feed inlet. Although the moving-bed system was successful for laboratory and some large-scale systems it has been found to suffer from difficulties in achieving the control of solid on a very large scale,

mass transfer efficiency loss due to uneven packing, attrition of the expansive packing, and low mobile phase velocity limited by the bed fluidization velocity. To overcome the problems in scale-up of the moving-bed system, the simulated moving-bed (SMB) process was developed.

In the SMB process the shifting of the feed and the product ports in the direction of fluid flow can simulate the movement of solids in the opposite direction, and the countercurrent contact of solid and liquid leads to a high mass transfer driving force. There have been two main approaches. The four-section SMB of the Sorbex Process, developed by UOP (4), consisted of a single column divided into a number of compartments, where the inlet and outlets in each compartment were controlled by using a master multiport valve. In Fig. 1(a), a four-section SMB is schematically presented and has four different flow zone as shown. In this run mode the flow rates of the withdrawn streams (extract and

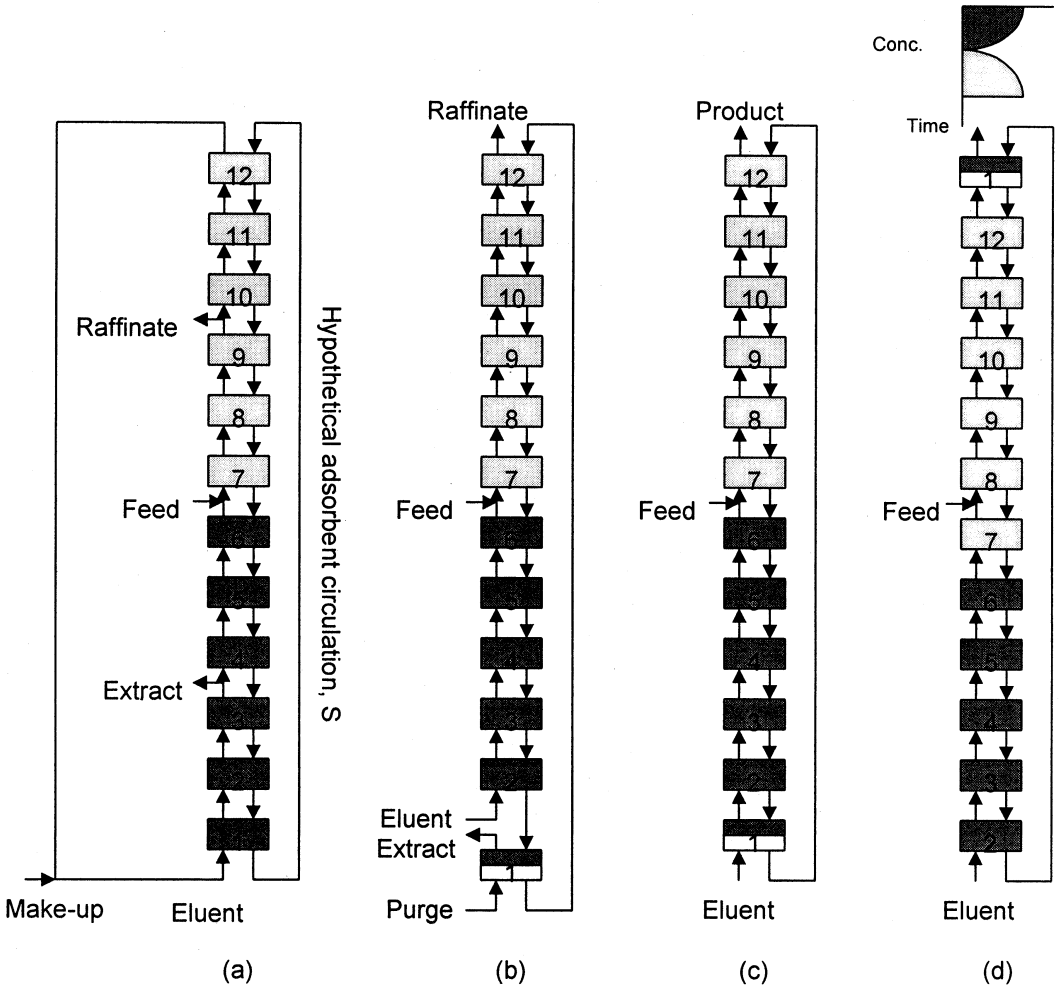

FIG. 1 Schematic explanation of SMB operation: (a) four-section SMB, (b) three-section SMB, (c) and (d) two-section SMB at the end and start of a one-switch interval.

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raffinate) were controlled accurately to maintain flow and pressure stability. In the three-section SMB (Fig. 1b), the column configuration was made up of three zones—an isolated purge, a prefeed, and a post feed section. The role of the isolated purge column is to elute the slowly moved component within the periodic time interval. This system can operate with or without recycling and can utilize all of the bed at any time. The four-section SMB operates isocratically while nonisocration in the three-section SMB is possible because to an isolated purge section. Ruthven et al. (5) gave a the useful and comprehensive review of these processes, and the advantages and disadvantages of two types of SMBs were well explained by Ganetsos et al. (6). Recently, SMB was developed into a SMBR (simulated moving-bed reactor) for carrying out chemical reaction and separation simultaneously. Especially in the case of reversible reactions, conversions exceed those of equilibrium (7, 8).

Various purge rates were tested with the same eluant and feed rate in a three-section SMB in previous work (9). It was successfully operated in these experiments without a high purge rate, and the sum of the eluant and feed rates was enough to purge the retained component within a switch time. When columns are shifted, the less adsorbed component moves through the purge column regenerated in the previous time interval and comes out one switch interval later. If the column configuration of a SMB can be modified as shown in Fig. 1(c) and (d), the products come out in the order of slow and fast moving-component due to the difference of exit time of each component. In this operation mode it is called a two-section SMB because there are only two different flow rate sections—prefeed and postfeed.

The object of this paper is to test the operation mode of a two-section SMB and compare its performance with that of a three-section SMB.

THEORETICAL

Two-section SMB operation is achieved by advancing the eluant and feed port at fixed time intervals by one column in the direction of fluid flow. The solid phase of the column is countercurrently moved to the direction of mobile phase flow at a fixed rate; that is, the slow-moving component can be made to travel with the solid phase and the fast-moving component with the mobile phase.

The essential requirement to achieve a good separation in such a process is that the flow rates in the two sections (prefeed and postfeed) must be adjusted in such a way as to achieve a net flow of the more strongly adsorbed species toward the prefeed section and a net flow of the less strongly adsorbed species toward the postfeed section as in Fig. 1(c) and (d). Because fructose is the slow-moving component and glucose is the fast-moving component in the cation exchanged resin (9), the flow ratio of the downward flow in the ad-

sorbed phase to the upward flow in the mobile phase [$\gamma = (1 - \varepsilon)Ku/\varepsilon v$] can be conveniently specified for a linear system.

$$\begin{aligned} &\text{Prefeed section:} && \gamma_F > 1.0, && \gamma_G < 1.0 \\ &\text{Postfeed section:} && \gamma_P > 1.0, && \gamma_G < 1.0 \end{aligned} \tag{1}$$

If the limiting constraints in two sections is satisfied by the same margin ($\alpha > 1.0$), Eq. (1) gives rise to a set of two simultaneous equations defining the relative flow rates throughout the system;

$$\begin{aligned} &\text{Prefeed section:} && E/S = K_G\alpha \\ &\text{Postfeed section:} && E/S + F/S = K_F/\alpha \end{aligned} \tag{2}$$

As each column contains the eluant phase in the void volume, its amount has to be compensated for by the eluant rate to get the actual eluant rate in the SMB unit.

$$E' = E + A\varepsilon u \tag{3}$$

MATHEMATICAL TREATMENT

In the SMB process, each adsorption column can be considered to be a fixed bed except at the moment of moving each inlet and outlet point. Since the plug flow model with a velocity-dependent overall mass transfer resistance well describes the chromatographic responses as represented by Lee et al. (9), the mass balance equations with the initial and boundary condition for each component and column can be given by Eqs. (4)–(7).

$$\frac{\partial c}{\partial t} = -v\frac{\partial c}{\partial z} - \frac{1-\alpha}{\alpha}k(v)(Kc - q) \tag{4}$$

$$\partial q/\partial t = k(v)(Kc - q) \tag{5}$$

$$\partial c/\partial z = 0, \qquad \text{at } z = L \tag{6}$$

$$c = q = 0, \qquad \text{at } t = 0 \tag{7}$$

When feed enters Column 7, the following conditions are written for the inlet points. At the eluent inlet point:

$$c_{1,0} = 0 \tag{8}$$

At the feed inlet point:

$$v_7c_{7,0} = v_6c_{6,1} + v_Fc_F \tag{9}$$

At the inlet points of other columns:

$$c_{j,0} = c_{j-1,1} \qquad (j = 2, \ldots, 6, 8, \ldots, 12) \tag{10}$$

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where the first subscript is the column number and the second subscript denotes either the inlet (0) and outlet (1) of the liquid stream in each column.

EXPERIMENT

The equipment used was the same as in previous work (9) and made up of a set of twelve 1-cm-diameter stainless steel columns with packed length of 30 cm (DOWEX 50W 12X in its Ca^{2+} form) linked alternately top and bottom to form a closed loop. The upper and lower bed supports consisted of two layer of fine mesh stainless steel screen. Two distributors with one inlet and 12 outlets were used to control feed and eluant inlet. All of columns were surrounded by a constant temperature enclosure. The countercurrent movement of a two-section SMB is simulated by sequencing a system of inlet and outlet port functions around 12 columns. In Fig. 1(c) the feed enters Column 7 and the less strongly adsorbed glucose moves with the eluant which enters the system at Column 1. The fructose, the strongly adsorbed component due to the formation of a chemical complex with Ca^{2+}, is preferentially retained by the resin. After one switch time, the position of the inlet and outlet port is advanced by one column as in Fig. 1(d). This simulation has the same effect as the movement of fructose with the stationary phase. Due to the difference of component velocity in the column and the column rearrangement, the strongly adsorbed fructose comes out first, followed by the less strongly adsorbed glucose. At the end of 12 such switches, one cycle is completed. After six cycles a pseudoequilibrium state can be reached.

Details of the six sets of experimental conditions are given at Table 1. These are satisfied by the flow conditions of Eq. (1) which define all the flow rate ratios for the two-section SMB unit. There are two equations and four variables (E, S, F, and α). Thus, by specifying two variables, the other variables can be

TABLE 1
Experimental Conditions

Run mode	Run no.	Eluant rate (cm^3/min)	Feed rate (cm^3/min)	Purge rate (cm^3/min)	Switch time (min)	Column length (cm)
Three-section SMB	1	3.6	0.72	4.32	3	30
	2	3.6	0.72	4.32	6	60
	3	3.6	0.72	4.32	9	90
Two-section SMB	4	3.6	0.72	—	3	30
	5	3.6	0.72	—	6	60
	6	3.6	0.72	—	9	90

obtained. In this work the switch time (thus the hypothetical solid adsobent flow rate) and the margin value (α) were chosen to make the degree of freedom zero. These experimental conditions are set for comparison of the performance of three- and two-section SMBs.

Prior to run the unit was fully purged by distilled water. At time zero the feed solution, containing 10 g/L of each sugar, was introduced at Column 7 as indicated in Fig. 1(c). Liquid samples were taken from the draw-off point of each column and analyzed by HPLC (Waters Associated Co.) using an "Aminex 87C" Column (Bio-Rad Co.). Elution was performed isothermally at 85°C with a constant fluid velocity of 0.4 cm^3/min. Under these conditions the retention time for the glucose and fructose peaks were 4.3 and 5.5 min, respectively. All runs of the two types of SMBs were carried out at 50°C and the linear isotherm range in which the components do not interact.

RESULTS AND DISCUSSION

Under the assumption that the components do not interact in the linear isotherm, the numerical solution of the transient model of each fixed bed was performed through the orthogonal collocation method (10, 11) which reduces the original partial differential equations to a set of ordinary ones. These differential equations were integrated through a fourth-order Runge–Kutta method (12), and selection of 10 collocation points at each fixed bed gave sufficient precision to calculate the theoretical profiles under the experimental conditions of this work. When columns were advanced at the end of a switch interval, concentration profiles corresponding to collocation points of each column were shifted by one column in the opposite direction of fluid flow according to the column movement. With this procedure, the pseudoequilibrium concentration profile can be calculated. In the numerical calculation, the equilibrium constants and overall mass transfer coefficients for glucose and fructose, which were determined independently from pulse response measurements in previous work (9), were used. They are summarized at Table 2.

TABLE 2
Parameters Used in This Work

	Glucose	Fructose
Adsorption equilibrium constant, K (—)	0.123	0.310
Velocity dependent effective overall mass transfer coefficient, $k(v)$ (min^{-1})	$3.155v/(v + 1.315)$	$2.941v/(v + 1)$

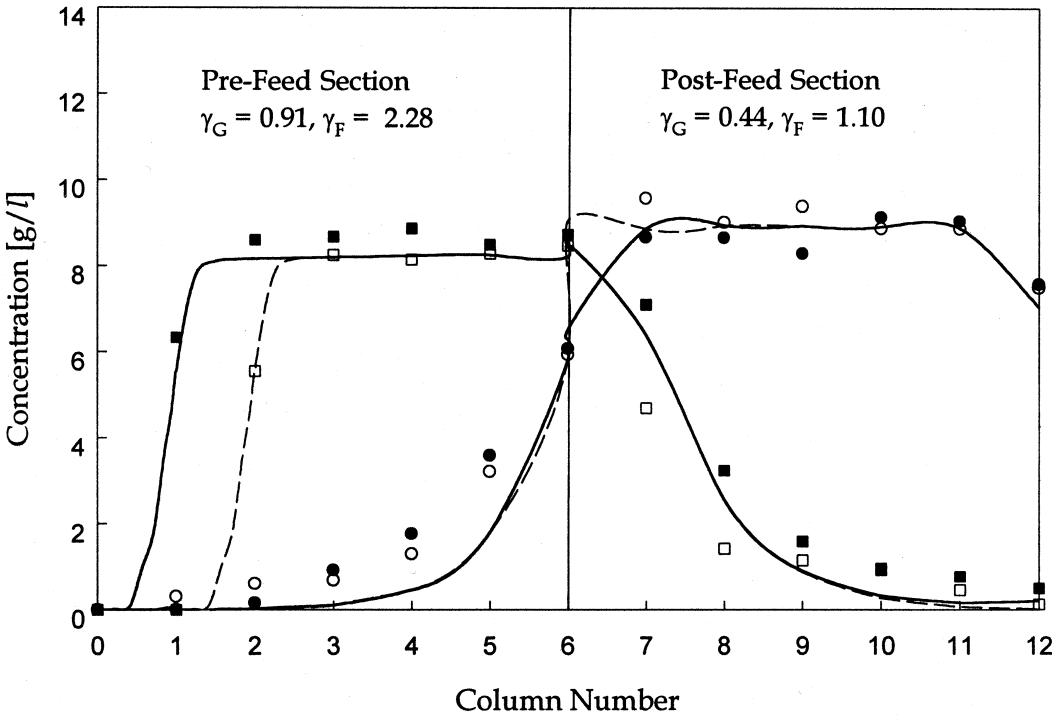

FIG. 2 Comparison of experimental and theoretical concentration profiles at the end of a switch time, total of 12 columns, each 30 cm. Three-section SMB: Run 1, cal, ⋯; exp, fru (□), glu (○). Two-section SMB: Run 4, cal, —; exp, fru (■), glu (●).

Representative on-concentration profiles are shown in Fig. 2 with the theoretical ones. In Runs 1 and 4 a total of 12 columns, each 30 cm, was used. It is evident that the theoretical curves provide a good representation of the experimental profiles. Some discrepancies between profiles stemmed from the fact that no allowance was made in the calculation for the solute hold-up in the valves and the intercolumn transfer tubes. In Runs 1 and 4, due to the similar zone of mass transfer, similar on-concentration profiles can be obtained.

In the simulated moving-bed system, it has been asked that how many columns are needed to simulate the moving-bed process. Some authors (13) showed that most of the benefit of counter current flow can be achieved by a rather modest degree of subdivision of the bed.

In the four-section SMB, the mean concentration profile averaged over the switch interval is more important rather than the profile at the midtime of the switch interval (14). If the bed was divided into very small subsections, one would have a perfect analogue of countercurrent flow. But if the subsection was decreased under the same total column length, the above two concentrations would have some difference. Because of the mass transfer effect and the

movement of concentration peaks with eluant flow, the product profiles decreased nonlinearly in the extract and increased in the raffinate during the switch interval.

On the other hand, in the three- and two-section SMBs, the product profiles were not continuously obtained with a switch time, and the question of the effect of subsections was how many were needed to get a high purity product. In each Runs 2 and 5 a total of six, each 60 cm, was used to study this effect with the same flow conditions of Run 1, and the experimental and calculated results are shown in Fig. 3. In this operation mode the column configuration of the three-section SMB was 1, 2, and 3 subsections in each purge, prefeed, and postfeed section, and that of the two-section SMB was 3 and 3 in the prefeed and postfeed sections. With the reduction of the prefeed zone in the three-section SMB, the on-column concentration profiles overlapped more. But in the two-section SMB, each two subsections were sufficient to separate the feedstock due to effective usage of the bed. When the subsection was reduced more, this effect was obviously apparent. In Runs 3 and 6 a total of four, each 90 cm, was used in each operation mode. As

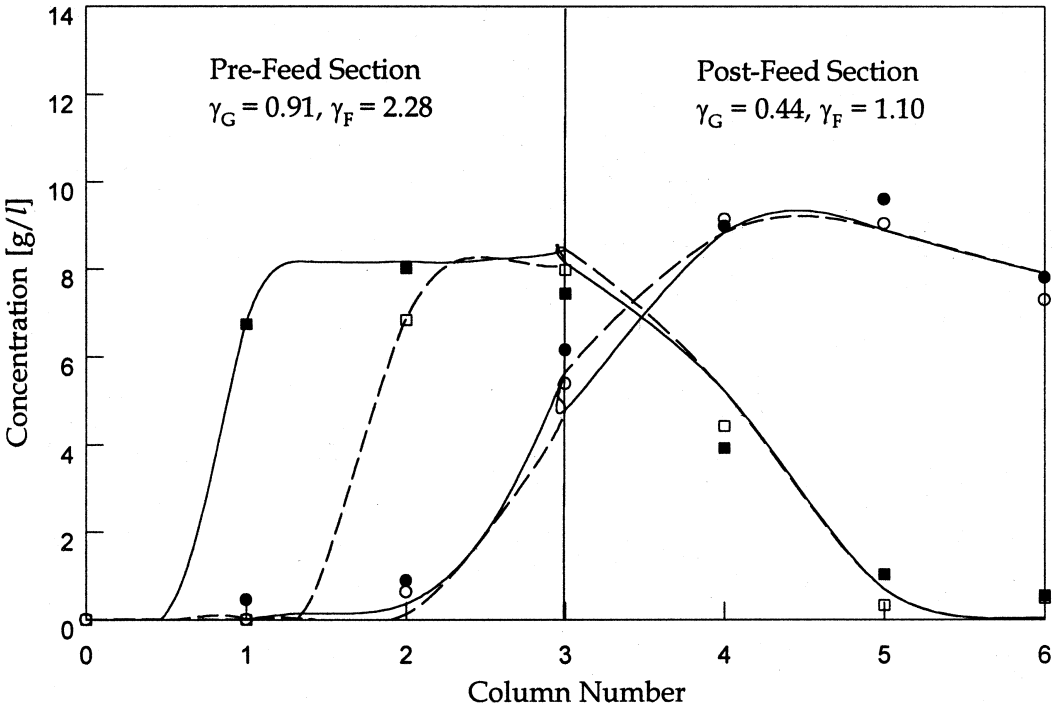

FIG. 3 Comparison of experimental and theoretical concentration profiles at the end of a switch time, total of 6 columns, each 60 cm. Three-section SMB: Run 2, cal, ⋯; exp, fru (□), glu (○). Two-section SMB: Run 5, cal, —; exp, fru (■), glu (●).

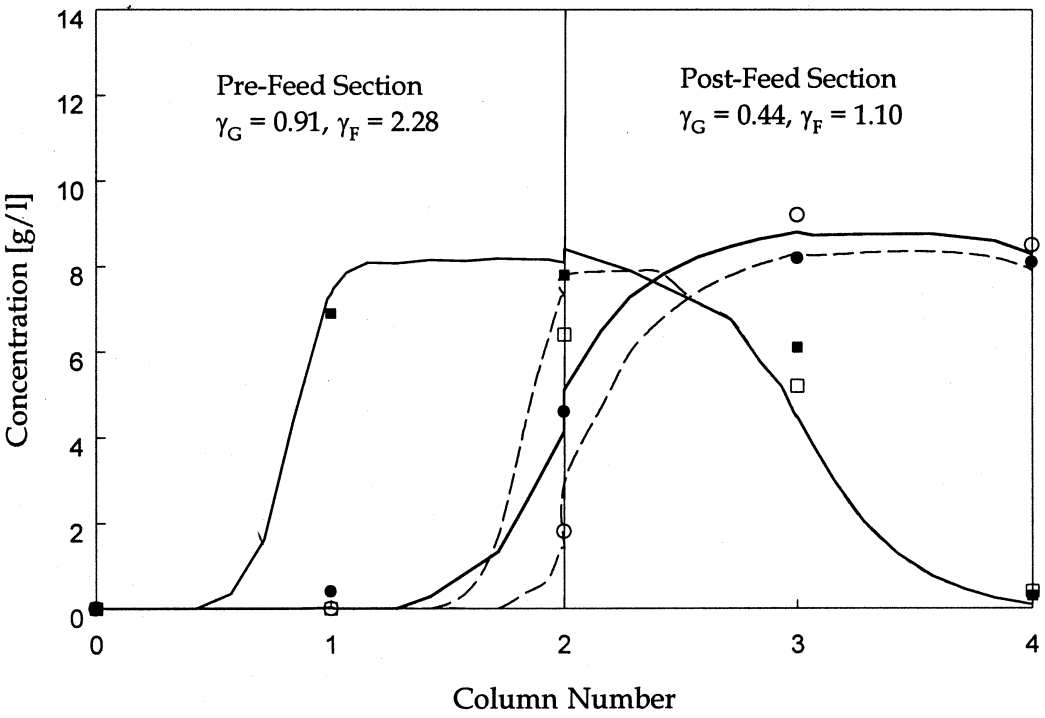

FIG. 4 Comparison of experimental and theoretical concentration profiles at the end of a switch time, total of 4 columns, each 90 cm. Three-section SMB: Run 3, cal, ⋯; exp, fru (□), glu (○). Two-section SMB: Run 6, cal, —; exp, fru (■), glu (●).

shown in Fig. 4, with a column configuration of 1, 1, and 2 in each zone of the three-section SMB, there was not sufficient bed length to separate the feed components continuously. In the "Sarex" type of process for glucose and fructose separation, wherein product purity requirements are modest, most units appear to be designed with either one or two subsections per section (15). In this respect, Run 6 corresponds to the operation mode of a four-section SMB with one subsection. In Figs. 5 to 7 the experimental concentration profiles of product are shown with the theoretical values. In (a) of all figures, all profiles are for a two-section SMB, and the two products exit from the last column of each column configuration. For a three-section SMB, fructose-rich products come out of a purge column and glucose-rich products come out of the last column. These profiles are represented in the same figures as (b). Here, the "critical" dilution was arbitrarily defined as that value where the product concentration was about 10% of the feed concentration. In all experiments the fructose-rich products above the "critical" dilution were collected and the glucose-rich products were collected from the start of its exit. The experimental results of this collection method are

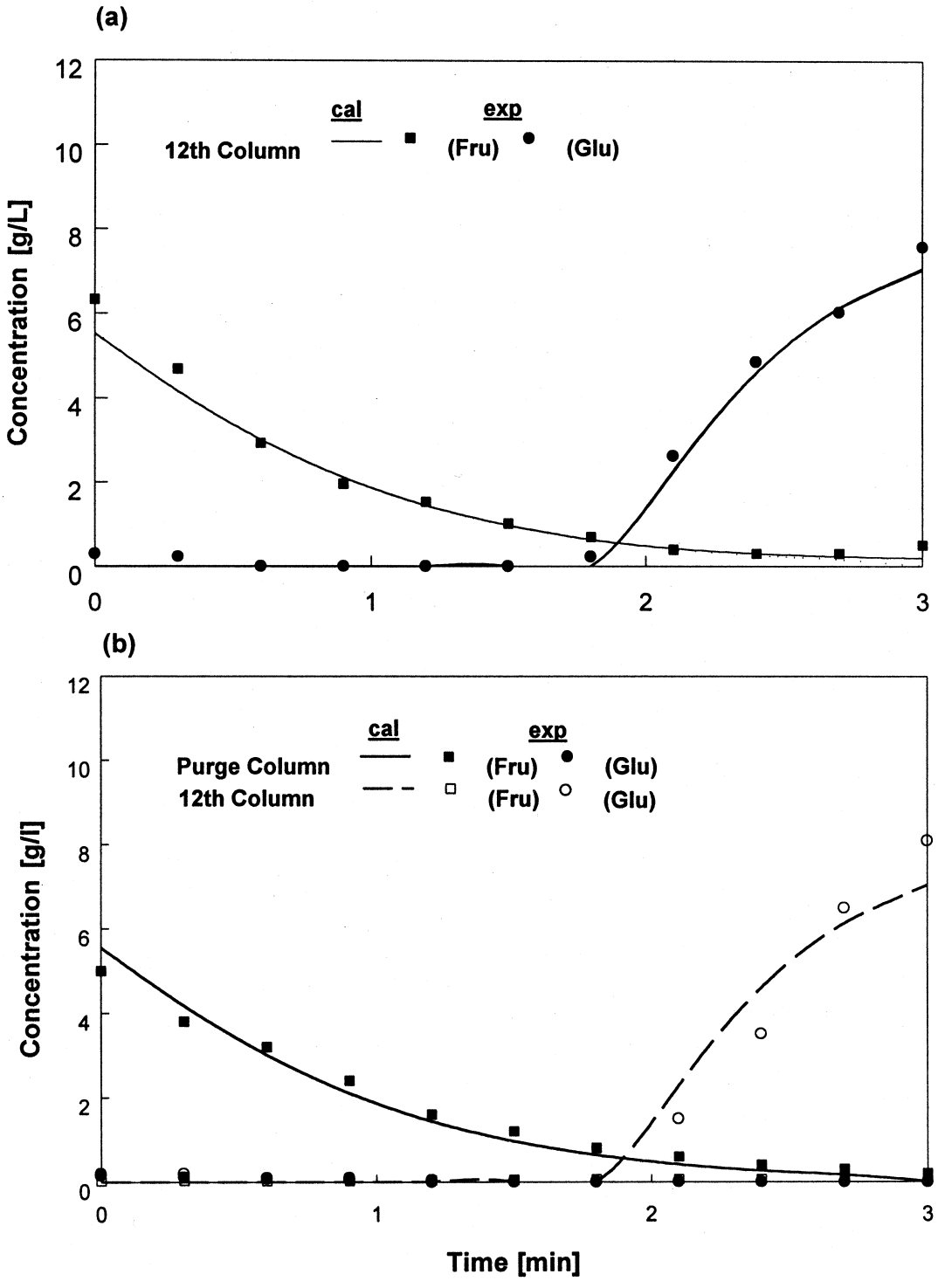

FIG. 5 Experimental and theoretical concentration profiles of fructose- and glucose-rich product: (a) two-section SMB (Run 4), (b) three-section SMB (Run 1).

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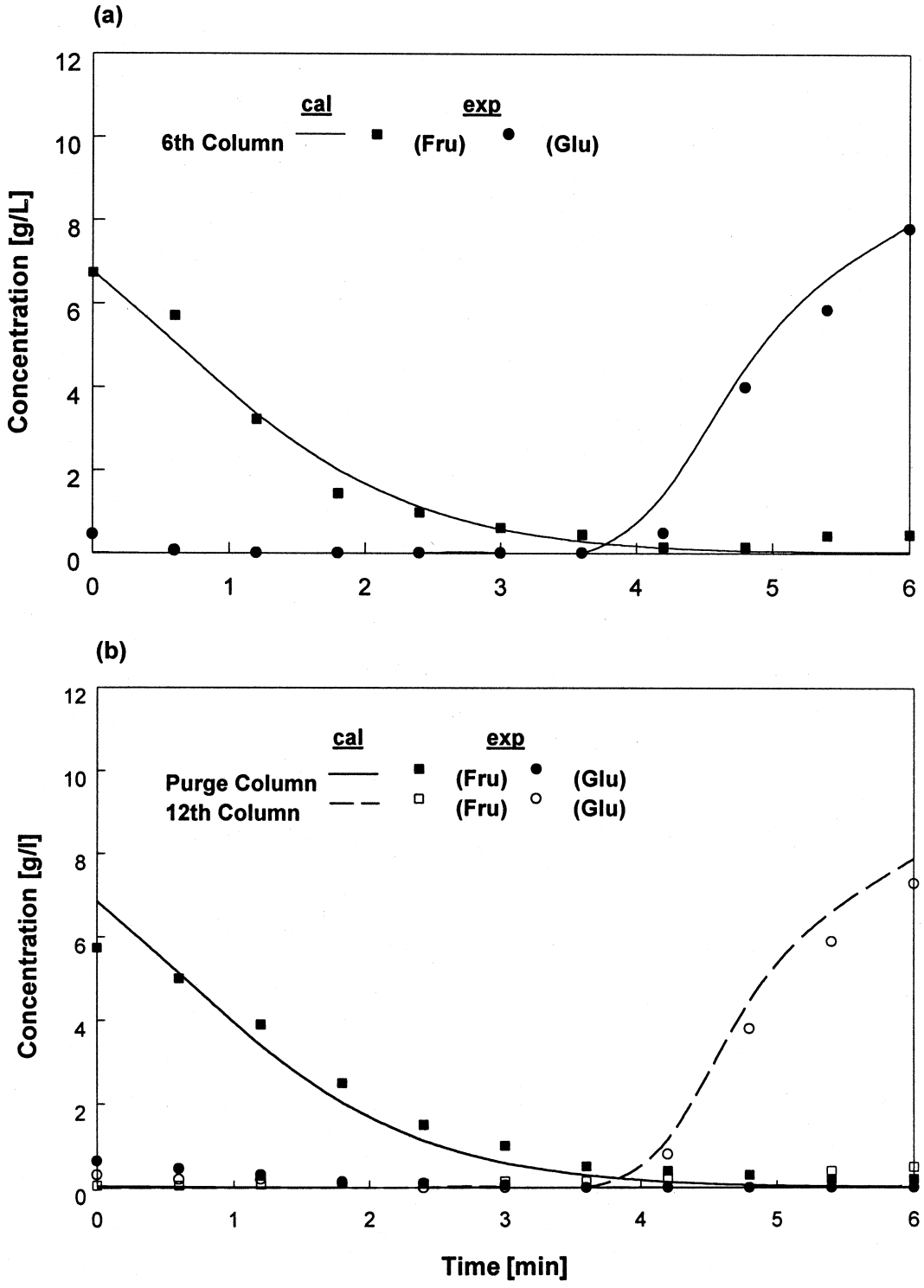

FIG. 6 Experimental and theoretical concentration profiles of fructose- and glucose-rich product: (a) two-section SMB (Run 5), (b) three-section SMB (Run 2).

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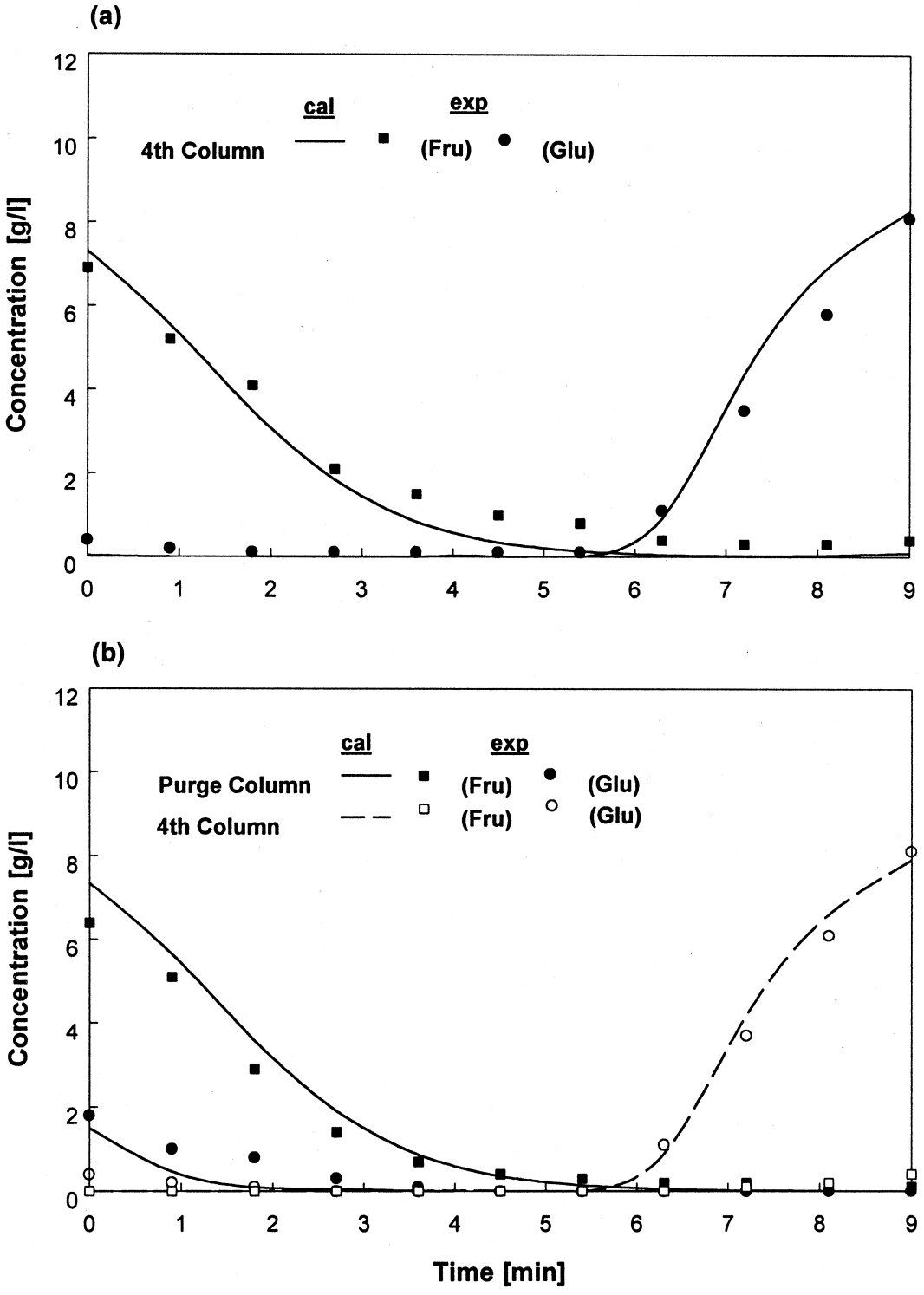

FIG. 7 Experimental and theoretical concentration profiles of fructose- and glucose-rich product: (a) two-section SMB (Run 6), (b) three-section SMB (Run 3).

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summarized in Tables 3 and 4, respectively. For the calculation of purity and recovery, the following relations were used;

$$\text{Purity of fructose} = \frac{\int_0^{t_c} C_F\, dt}{\int_0^{t_c} (C_F + C_G)dt}$$

$$\text{Purity of glucose} = \frac{\int_{t_s}^{\tau} C_G\, dt}{\int_{t_s}^{\tau} (C_F + C_G)dt} \tag{11}$$

$$\text{Recovery of fructose} = \frac{v_F \int_0^{t_c} C_F\, dt}{v_F c_F \tau}$$

$$\text{Recovery of glucose} = \frac{v_G \int_{t_s}^{\tau} C_G\, dt}{v_F c_F\, \tau} \tag{12}$$

TABLE 3
Experimental Results in the Fructose-Rich Product[a]

Run mode	Run no.	Product collection period (min)	Average concentration (g/L)	Purity (%)	Recovery (%)
Three-section SMB	1	0 to 1.5	2.82 (2.79)	96.57 (100)	84.60 (83.70)
	2	0 to 2.4	3.75 (3.64)	92.29 (99.67)	90.10 (87.27)
	3	0 to 3.6	3.24 (3.80)	80.93 (91.87)	77.80 (91.27)
Two-section SMB	4	0 to 15	2.96 (2.79)	97.35 (100)	88.71 (88.82)
	5	0 to 2.4	3.59 (3.74)	97.98 (100)	86.04 (89.66)
	6	0 to 3.6	3.90 (3.73)	96.60 (99.91)	93.67 (89.60)

[a] Values in parentheses are the theoretical results.

TABLE 4
Experimental Results in the Glucose-Rich Product[a]

Run mode	Run no.	Product collection time (min)	Average concentration (g/L)	Purity (%)	Recovery (%)
Three-section SMB	1	1.8 to 3	3.89 (4.13)	98.24 (100)	93.30 (98.60)
	2	3.6 to 6	3.54 (4.04)	92.06 (99.57)	84.90 (96.96)
	3	5.4 to 9	3.74 (3.90)	96.76 (99.99)	89.70 (93.60)
Two-section SMB	4	1.8 to 3	4.36 (4.13)	88.71 (92.39)	104.64[b] (99.00)
	5	3.6 to 6	3.89 (4.25)	92.73 (97.47)	93.36 (100.00)
	6	5.4 to 9	3.63 (4.07)	90.06 (98.69)	87.00 (97.80)

[a] Values in parentheses are the theoretical results.
[b] Values too high due to analytical inaccuracy.

where t_s is the starting time of the glucose exit and t_c is when the critical concentration collection ends.

From the results of experiment and calculation, the two-section SMB can be characterized as follows compared with the three-section SMB:

1. It is more economic based on the cost of apparatus and operation
2. It uses the mobile phase more effectively because it does not use the excess purge flow rate
3. It uses the bed more effectively and therefore it can reduce the subsection without a loss of purity

CONCLUSION

A new operation mode using a two-section simulated moving bed (SMB) was developed through modification of the three-section SMB. The performances of the two- and three-section SMB processes were compared through application of the continuous separation of glucose and fructose.

Although general conclusions cannot be drawn from the limited range of experimental and theoretical results, the two-section SBM process shows the same performance as the three-section SMB process in obtaining high fruc-

tose corn syrup (55 to 90 w/w% fructose). In the run mode with reduced subsections, it used the bed more effectively so it obtained a fructose-rich product with higher purity than did a three-section SMB.

NOTATIONS

A	cross-sectional area of column (cm^2)
c	fluid-phase concentration ($g \cdot L^{-1}$)
c_F	feed concentration ($g \cdot L^{-1}$)
E	eluant flow rate ($cm^3 \cdot min^{-1}$)
E'	actual eluant flow rate ($cm^3 \cdot min^{-1}$)
F	feed flow rate ($cm^3 \cdot min^{-1}$)
K	adsorption equilibrium constant ($= q^*/c$)
k	effective overall mass transfer coefficient (min^{-1})
L	length of adsorbed bed (cm)
q	adsorbed phase concentration ($g \cdot L^{-1}$)
S	hypothetical adsorbent recirculation rate in equivalent countercurrent system [$= A(1 - \varepsilon)u$] ($cm^3 \cdot min^{-1}$)
SMB	simulated moving bed
t	time (min)
u	hypothetical solid velocity ($= L/\tau$) (cm/min)
v	interstitial fluid phase velocity (cm/min)
v_f	feed velocity (cm/min)

Greek Letters

α	margin defined in Eq. (2)
γ	dimensionless parameter [$= (1 - \varepsilon)Ku/\varepsilon v$]
ε	void fraction of packed bed
τ	switch time (min)

Subscript

F	fructose
G	glucose

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