Comparative Study of Flow Schemes for a Simulated Countercurrent Adsorption Separation Process

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Continuous separation of three carbohydrate mixtures [fructose-dextran (M.W. ≈ 9,400), raffinose-dextran (M.W. ≈ 6,000), and fructose-raffinose] has been carried out using a simulated countercurrent process with silica gel as the sorbent and deionized water as the eluent. Experiments were conducted using the commercial four-section arrangement and a modified three-section flow scheme which does not recycle part of the eluent for reuse. Data for the four-section flow scheme reflect a much higher product concentration in the raffinate and a substantially lower rate of fresh eluent consumption than for the three-section scheme. It is concluded that when both the extract and raffinate products are desired, the four-section scheme should be used for the separation of a mixture with a small separation factor which usually requires a large quantity of eluent to achieve an efficient separation. The behavior of the simulated countercurrent process was simulated theoretically by representing the process in terms of an equivalent true countercurrent system. A good fit to the experimental data was obtained when the process was configured in the four-section mode. However, the same theoretical approach failed to provide a good representation of the behavior of the three-section scheme in one respect: predicted concentrations of the raffinate product were about two times higher than experimental values. The discrepancy may be attributed to the periodic switching of fluid inlet and outlet points in the operation of the three-section simulated countercurrent unit.

Introduction

Simulated countercurrent separation processes have achieved extensive applications in the petrochemical and sugar industries over the years. These commercial processes are marketed by UOP (U.S.A.) (Broughton, 1968; Broughton et al., 1970), Toray Industries, Inc. (Japan) (Otani, 1973), and Mitsubishi Chemical Industries Ltd (Japan) (Hirota et al., 1981). The process is designed to mimic the behavior of a true countercurrent process depicted schematically in Figure 1 for the separation of a mixture containing A and B, with component A being the more strongly adsorbed species. Figure 1 shows that the adsorbent is circulated continuously in a closed cycle and moves down the adsorbent bed from top to bottom. The feed and eluent enter continuously and flow upward, countercurrently to the adsorbent flow. The extract stream which contains the more strongly adsorbed component and the raffinate stream which contains the less strongly adsorbed component, each mixed with the eluent, are withdrawn continuously from the system. The four streams of liquid introduction and withdrawal effectively divide the system into four separate sections, each of which performs a different function. Section I, bounded by the eluent inlet and extract withdrawal, is a desorption section. The eluent entering at the bottom of this section removes the adsorbed A from the descending adsorbent. A fraction of the liquid leaving the top of this section is withdrawn as the extract stream while the remainder flows upward into section II. Section II lies between the extract withdrawal and feed inlet and serves to desorb B from the adsorbent which carries both A and B as it has been in contact with the fresh feed before entering this section. In section III, located between

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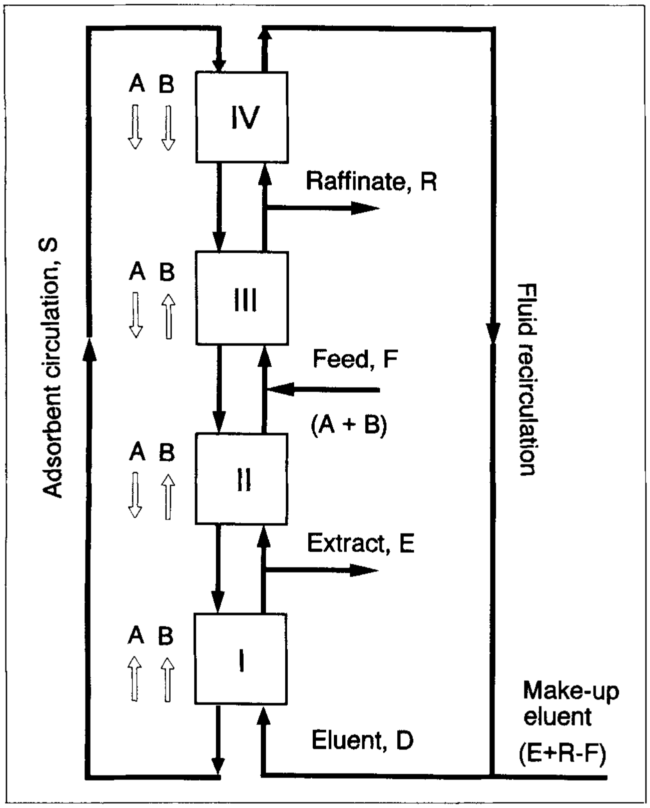

Figure 1. Four-section equivalent true countercurrent process.

The arrows labeled with A and B indicate the direction of the net flow of solutes A and B, respectively.

the feed inlet and raffinate withdrawal, A is transferred from the rising liquid stream to the downward moving adsorbent so that the liquid emerging at the top of this section contains only B. A portion of this liquid is withdrawn as the raffinate stream. Finally, section IV, situated between the raffinate withdrawal and eluent inlet, is used to desorb B from the rising liquid by the descending adsorbent. The liquid flowing out of this section should ideally contain pure eluent only which is recirculated to the eluent inlet point for reuse.

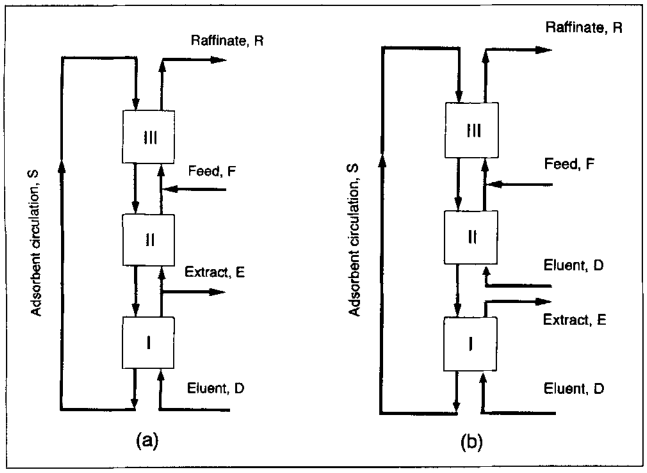

Figure 2. Three-section flow schemes.

Due to the difficulties and complications associated with solid circulation, commercial processes simulate the solid movement in Figure 1 by advancing the liquid inlet and outlet points at fixed time intervals through a fixed bed in the direction of liquid flow. The four-section flow scheme depicted in Figure 1 is used by all the currently available commercial processes which differ in the design of equipment configuration to effect the countercurrent movement rather than in the basic concept. For example, the sequential movement of fluid entry and exit points along a single adsorbent chamber in processes developed by UOP is controlled by a complex rotary valve while processes developed by Toray Industries consist of a series of packed columns in which fluid inlet and outlet points are switched periodically by using a sequence of on-off valves.

The four-section flow scheme has been extensively studied in the open literature in recent years (see, for example, Hashimoto et al., 1983; Ching et al., 1985; Kubota et al., 1989; Ernst and Hsu, 1989). Two variations of this commercial flow scheme, which are shown in Figure 2, have also been described. The function of each individual section in these three-section schemes is identical to the corresponding section in the four-section process. These three-sections schemes differ in the arrangement of section I which, in the case of Figure 2b, is isolated for purging. It is obvious that the product recovered from the extract in such a process (Barker and Ching, 1980; Ching and Ruthven, 1984; Barker et al., 1990) is considerably more diluted than that recovered from the process depicted in Figure 2a (Szepesy et al., 1975; Hashimoto et al., 1989). Hence, in this study the process depicted in Figure 2a (hereafter referred to as the three-section scheme) was selected as the representative of the two flow schemes.

When a separation process requires the use of an expensive eluent, the four-section scheme has the obvious advantage over the three-section process with respect to the consumption of fresh eluent, that is, section IV allows the recycling of part of the eluent for reuse. However, there are no comparative performance data in the literature for these two flow schemes. In view of the recent development of commercial simulated countercurrent processes which are being adapted for bioseparations (Johnson and Kabza, 1990), a question naturally arises as to how one can choose the most appropriate process configuration for a particular separation. The major goal of this study was therefore to explore this question in some detail. Continuous separation of binary carbohydrate mixtures was investigated using a laboratory-scale simulated countercurrent unit which was configured in both the commercial four-section arrangement and the three-section flow scheme. In order to compare the flow schemes in a meaningful way, we assumed that the aim of the separation was to produce valuable products in both the extract and raffinate streams.

Experimental

The general arrangement of the experimental system, which is shown schematically in Figure 3, has been described previously (Ching et al., 1991). A set of eight identical jacketed columns, each 47.5 cm × 1.4 cm ID, was packed with silica gel (particle size = 22 μm, pore diameter = 66 Å). The columns were connected in series and each column was equipped with three inlet lines (feed, eluent, and transfer from the preceding column) and four outlet lines (extract, raffinate, recycle, and

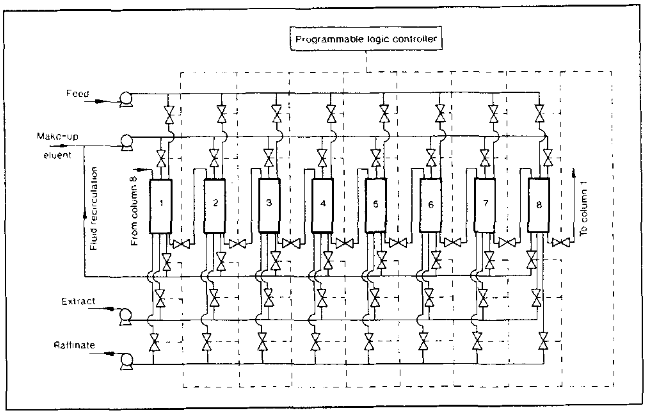

Figure 3. Physical arrangement of columns and switch valves in the four-section simulated countercurrent unit.

transfer to the next column) which were controlled by solenoid valves. The valves were operated in an appropriate sequence by a programmable logic controller. Simulated countercurrent operation was achieved by advancing the feed, eluent, extract, raffinate, and recirculation points by one column, at fixed time intervals, in the direction of liquid flow. At the start of each experiment all columns were purged with water until steady flow rates of all inlet and outlet lines were established. The feed line was then switched to a solution containing carbohydrates. The flow rates of feed and eluent were controlled by HPLC pumps while those of extract and raffinate were controlled by peristaltic pumps. All runs were carried out at 25°C and liquid samples were withdrawn from sampling points located at the outlet of each column and analyzed by liquid chromatography. In addition, cumulative samples of the extract and raffinate streams were collected and analyzed in the same way. Operation was continued for several cycles until the concentration profile showed no significant change between successive cycles.

The experimental unit was first configured in the four-section flow scheme with two columns in each section. The unit was used to separate three different carbohydrate mixtures, each containing two solutes dissolved in deionized water: (1) fructose-dextran T9 (M.W. ≈ 9,400), (2) raffinose-dextran T6 (M.W. ≈ 6,000), and (3) fructose-raffinose. It was then configured in the three-section mode with two columns in section I, two in section II, and four in section III to separate the same set of binary mixtures. Hence, a total of six runs was carried out.

The adsorption equilibrium constants and effective mass-transfer coefficients for fructose, raffinose, dextran T6, and dextran T9 were measured by pulse chromatography using an independent column (30 cm × 1.1 cm ID) packed with the same adsorbent. Similarly, the average dispersion characteristics and bed voidage of the countercurrent unit were determined from the retention times of D_2O and blue dextran respectively using four representative columns isolated from the rest of the system.

Table 1. Equilibrium and Kinetic Parameters at 25°C

	K	k (min^{-1})
Fructose	0.69	5.52
Raffinose	0.56	3.42
Dextran T6	0.23	2.84
Dextran T9	0.13	0.60

Results and Discussion

The response peaks obtained from pulse chromatographic experiments carried out over a range of liquid velocities with an independent column were analyzed according to the method of moments to yield the equilibrium and kinetic parameters (Ruthven, 1984). Table 1 summarizes the results for the four carbohydrates. The bed voidage of the countercurrent unit was also determined from pulse chromatographic experiments using blue dextran, a molecule too large to penetrate the adsorbent pores, as tracer. The average bed voidage of four representative columns was found to be 0.45. Similarly, pulse experiments performed on the four columns using D_2O as tracer measured the axial mixing in the columns as well as in the holdup of the column end sections and the pipes and valves between the columns. We assume that all these mixings can be accounted for by an effective axial dispersion coefficient D_L which depends on liquid velocity. The average dispersion coefficient for the packed columns is given by $D_L = 0.16v$. It can be seen that D_L is directly proportional to the liquid interstitial velocity v. In turn, we assume the axial dispersion coefficient for the simulated countercurrent unit to be given by $D_L = 0.16(u + v)$ since the effective liquid flow rate in each section of the countercurrent unit is $(u + v)$. These equilibrium, mass transfer, column dispersion and bed voidage values were required as input parameters for the theoretical model developed to analyze the behavior of the countercurrent unit.

Operation of the simulated countercurrent unit may be conveniently understood by considering the equivalent true countercurrent process, as shown in Figures 1 and 2. The essential requirement to achieve separation in such a process is that the flow rates in each section must be adjusted in such a way as to achieve a net flow of the more strongly adsorbed species toward the extract point, and a net flow of the less strongly adsorbed species toward the raffinate point. For a linear system these flow constraints together with a knowledge of the adsorption equilibrium constants allow one to define all the flow rates throughout the system. The procedure for obtaining the desired operating conditions from these considerations has been discussed in detail elsewhere (Ching et al., 1985). Six experimental runs were carried out under the flow conditions given in Table 2. Dilute feed was used in all cases to ensure linear behavior.

The resulting steady state concentration profiles, measured at the midpoint of the switch interval, are shown in Figures 4-6. Also shown in these figures are theoretical profiles calculated from the previously developed continuous countercurrent dispersed plug flow model with a linear driving force rate expression (Hidajat and Ching, 1990). The behavior of the simulated countercurrent system was modeled by considering the hypothetical equivalent true countercurrent processes sketched in Figures 1 and 2. Details of the numerical solution of the model equations by the method of orthogonal collocation have been given by Hidajat and Ching (1990). It is

Table 2. Summary of Experimental Conditions

Feed Flow Scheme	Fructose (5%) + Dextran T9 (5%) Four-Section	 Three-Section	Raffinose (5%) + Dextran T6 (5%) Four-Section	 Three-Section	Fructose (5%) + Raffinose (5%) Four-Section	 Three-Section
Feed, F (cm^3/min)	2.0	2.0	1.0	1.0	0.5	0.5
Solid, S (cm^3/min)	4.1	4.1	3.9	3.9	6.2	6.2
Eluent, D (cm^3/min)	3.1	3.1	2.4	2.4	4.4	4.4
Extract, E (cm^3/min)	2.5	2.5	1.4	1.4	0.8	0.8
Raffinate, R (cm^3/min)	2.1	2.6	1.2	2.0	0.8	4.1
Actual Eluent* (cm^3/min)	6.5	6.5	5.5	5.5	9.5	9.5
Switch Time** (min)	9.8	9.8	10.3	10.3	6.5	6.5

*Actual eluent = $[\epsilon/(1-\epsilon)]S + D$ where $\epsilon = 0.45$.

**Switch time = $[(1-\epsilon)aL]/S$ where $a = 1.54$ cm^2 and $L = 47.5$ cm.

evident that the model provides satisfactory representations of the experimental profiles for the four-section process arrangement. This observation is in agreement with those of previous workers (Hashimoto et al., 1983; Kubota et al., 1989). Considering that all the model parameters were independently determined, one may conclude that this simplified model can provide useful guidance in the design and optimization of a simulated countercurrent process. However, the same theoretical approach failed to provide a good representation of the behavior of the three-section scheme in one respect: predicted concentrations of the raffinate product were about two times higher than experimental values. The discrepancy may be attributed to the addition of a "clean" column to the top of section III at every switch time. Consequently, the raffinate product recovered from section III would be diluted by the interstitial eluent contained in the "clean" column. The extent of dilution may be estimated from actual and hypothetical eluent flow rates which are described by Eqs. 1 and 2, respectively (the actual eluent flow refers to the effective flow rate of eluent in the simulated countercurrent unit while the hypothetical eluent flow refers to the flow rate of eluent in the equivalent true countercurrent process).

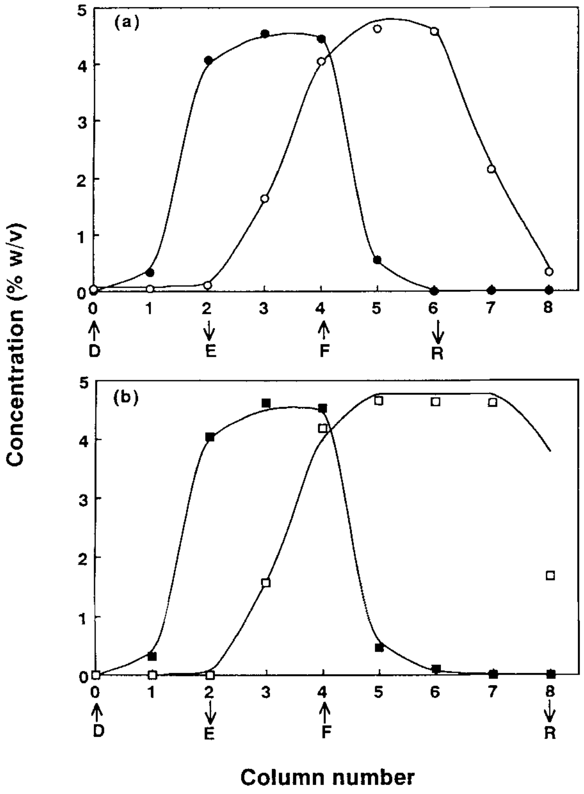

Figure 4. Comparison of experimental (points) and theoretical (curves) concentration profiles for the separation of fructose from dextran T9: (a) four-section operation, fructose (•) and dextran T9 (○); and (b) three-section operation, fructose (■) and dextran T9 (□).

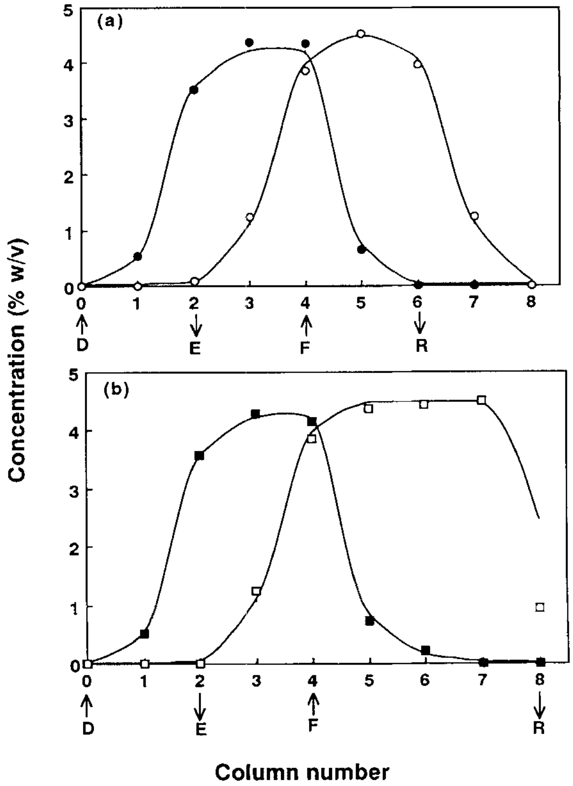

Figure 5. Comparison of experimental (points) and theoretical (curves) concentration profiles for the separation of raffinose from dextran T6: (a) four-section operation, raffinose (•) and dextran T6 (○); and (b) three-section operation, raffinose (■) and dextran T6 (□).

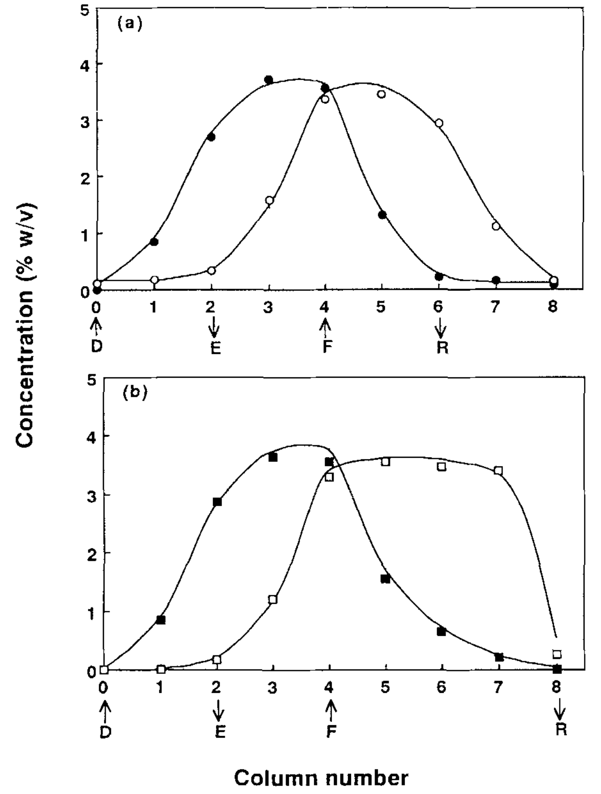

Figure 6. Comparison of experimental (points) and theoretical (curves) concentration profiles for the separation of fructose from raffinose: (a) four-section operation, fructose (•) and raffinose (○) and (b) three-section operation, fructose (■) and raffinose (□).

$$\text{Actual eluent flow} = D + \left[\frac{\epsilon}{1-\epsilon}\right] S \tag{1}$$

$$\text{Hypothetical eluent flow} = D \tag{2}$$

Dividing Eq. 1 by Eq. 2 we get:

$$\frac{\text{Actual eluent flow}}{\text{Hypothetical eluent flow}} = 1 + \left[\frac{\epsilon}{1-\epsilon}\right] \frac{S}{D} \tag{3}$$

The ratio on the left-hand side of Eq. 3, calculated using the numerical values of S and D listed in Table 2 for the three-section runs, is approximately equal to two. It is similar to the ratio of theoretical to experimental raffinate product concentrations (see Figures 4b, 5b, and 6b). Eq. 3 therefore gives an approximate indication of the deviation between model predictions and experimental data. Based on the reason given above, the discrepancy is also expected to be present in the analysis of the four-section scheme. However, the phenomenon of product dilution was not observed in the four-section scheme because the liquid stream leaving section IV contained essentially pure eluent. In other words, the addition of a "clean" column in this section has no noticeable effect on the "concentration" of this liquid stream. It should be noted at this point that the discrepancy between predicted and experimental data for the three-section flow scheme has not been reported in the past. A plausible explanation may be that in previous studies the raffinate product was always recovered in low concentrations and consequently it was difficult to distinguish the discrepancy from experimental measurement uncertainties. This is aptly illustrated by Figure 6b which represents the case of recovering the raffinate product in low concentration.

It is worth mentioning that the number of subdivisions of the adsorbent bed did not play a role in the failure of the three-section scheme to give an adequate approach to the behavior of the equivalent true countercurrent process. The question of how many subdivisions of the adsorbent bed are needed for a simulated countercurrent unit to approach true continuous countercurrent behavior has been discussed in detail by Hidajat et al. (1986). Obviously if the bed were divided into very small subsections one would have a perfect analogue of countercurrent flow. The good agreement between the experimental results and model predictions for the four-section and three-section (except for the raffinate product) schemes indicates that the behavior of the present unit with eight subdivisions of the bed was fairly close to true countercurrent behavior. Therefore, an increase in the number of subdivisions of the bed is unlikely to reduce the difference between predicted and experimental values of the raffinate product concentration for the three-section scheme. Equation 3 clearly indicates that the dilution of the raffinate product concentration is due to the intrinsic nature of the operation of the simulated countercurrent unit.

Table 3 shows the experimental results of the extract stream expressed in terms of product purity, recovery, and concentration ratio. It is evident that in the separations of both the

Table 3. Summary of Extract Purities, Recoveries, and Concentration Ratios Obtained from the Four-Section and Three-Section Operations

Feed	Fructose + Dextran T9		Raffinose + Dextran T6		Fructose + Raffinose	
Flow Scheme	Four-Section	Three-Section	Four-Section	Three-Section	Four-Section	Three-Section
Concentration (% w/v)	Fr = 4.06 T9 = 0.10	Fr = 4.05 T9 = 0.00	Ra = 3.52 T6 = 0.10	Ra = 3.57 T6 = 0.00	Fr = 2.69 Ra = 0.34	Fr = 2.91 Ra = 0.17
Purity	0.98	1.00	0.97	1.00	0.89	0.94
Recovery	1.00	1.00	0.99	1.00	0.86	0.93
Concentration Ratio	0.81	0.81	0.70	0.71	0.54	0.58

Extract purity is defined as $c_i/(c_i + c_j)$ where i is the more strongly adsorbed species and j is the less strongly adsorbed species. Extract recovery is defined as the ratio of amount of i recovered in the extract to initial amount of i in feed. Extract concentration ratio is defined as the ratio of concentration of i in the extract to initial concentration of i in feed.

Table 4. Summary of Extract Purities, Recoveries, and Concentration Ratios Obtained from the Four-Section and Three-Section Operations

Feed Flow Scheme	Fructose + Dextran T9		Raffinose + Dextran T6		Fructose + Raffinose	
	Four-Section	Three-Section	Four-Section	Three-Section	Four-Section	Three-Section
Concentration (% w/v)	Fr = 0.00 T9 = 4.58	Fr = 0.00 T9 = 1.68	Ra = 0.00 T6 = 3.97	Ra = 0.00 T6 = 1.05	Fr = 0.25 Ra = 2.89	Fr = 0.00 Ra = 0.26
Purity	1.00	1.00	1.00	1.00	0.92	1.00
Recovery	0.96	1.00	0.95	1.00	0.92	0.96
Concentration Ratio	0.92	0.34	0.79	0.21	0.58	0.05

Raffinate purity is defined as $c_j/(c_i+c_j)$ where i is the more strongly adsorbed species and j is the less strongly adsorbed species. Raffinate recovery is defined as the ratio of amount of j recovered in the raffinate to initial amount of j in feed. Raffinate concentration ratio is defined as the ratio of concentration of j in the raffinate to initial concentration of j in feed.

fructose-dextran T9 and raffinose-dextran T6 mixtures, very high levels of purity and recovery were obtained. There are virtually no differences between the results of the four-section and three-section flow schemes. In contrast, in the case of separating a mixture containing fructose and raffinose, the three-section flow scheme produced higher levels of extract purity and recovery than the four-section scheme. This difference in performance may be explained in terms of the separation factor which is defined as the ratio of the equilibrium constants for the more strongly adsorbed species and the less strongly adsorbed species. The separation factors for the three binary mixtures are 5.30 (fructose-dextran T9), 2.43 (raffinose-dextran T6), and 1.23 (fructose-raffinose). The experimental results indicate that the existing simulated countercurrent unit was capable of producing a complete separation of a mixture with a separation factor of 2.43 or higher. It is however clear that the experimental unit, configured in either the four-section scheme or the three-section arrangement, could not perform a complete separation of the mixture with a separation of 1.23. One consequence of this incomplete separation in the four-section scheme is that the liquid stream leaving section IV, which was to be recycled to section I after mixing with fresh make-up eluent, carried a certain amount of the less strongly adsorbed species. This amount of the less strongly adsorbed species subsequently showed up in the extract which was withdrawn from the top of section I. Hence, section IV was found to have a detrimental effect on the purity of the extract when the simulated countercurrent unit failed to provide an efficient separation of a mixture with a small separation factor.

Table 4 shows that higher levels of purity and recovery of the raffinate product were also obtained when the fructose-raffinose mixture was separated using the three-section scheme. The reason for this improvement has already been discussed, that is, an additional amount of raffinose, which would have been recycled to section I in the four-section scheme due to incomplete desorption in section IV, was instead recovered in the raffinate stream of the three-section scheme. However, the benefits of including section IV in the simulated countercurrent process can be seen in the concentration of the raffinate product and the consumption rate of fresh eluent. Table 4 shows that the raffinate products were considerably diluted when separations were carried out without the use of section IV. Data for the separation of fructose from raffinose show that the raffinate product concentration in the four-section operation was about ten times that of the three-section operation. This is undesirable since costly evaporation steps are then required to obtain the raffinate product at the desired concentration. Comparisons of the consumption rate of flesh eluent for the four-section and three-section flow schemes are shown in Table 5. As expected, the three-section scheme consumed much more fresh eluent than the four-section scheme. The consumption rate increases with decreasing value of the separation factor. It is clear that in the separation of a mixture with a small separation factor, the benefits derived from the four-section scheme in terms of a relatively higher raffinate product concentration and a lower rate of eluent consumption far outweigh the benefits of minor improvement in product purity and recovery that may be obtained from a flow scheme without section IV.

Conclusions

Experiments were carried out to generate comparative performance data for a simulated countercurrent unit configured in both the four-section and three-section flow schemes. It is observed that in the separation of a mixture with a small separation factor, the four-section flow scheme generated significant savings in terms of eluent consumption and processing costs associated with subsequent evaporation steps to produce the desired raffinate product concentration. However, the four-section scheme produced a lower extract product purity in

Table 5. Comparisons of Fresh Eluent Consumption Rate for the Four-Section and Three-Section Operations

Feed Flow Scheme	Fructose + Dextran T9		Raffinose + Dextran T6		Fructose + Raffinose	
	Four-Section	Three-Section	Four-Section	Three-Section	Four-Section	Three-Section
Eluent Consumption (cm^3/min)	2.6	6.5	1.6	5.5	1.1	9.5
Consumption Ratio*	2.5		3.4		8.6	

* Defined as the ratio of eluent consumption for the three-section operation to eluent consumption for the four-section operation.

comparison to the three-section scheme when the experimental unit failed to perform a complete separation. It is shown that the behavior of the four-section flow scheme could be well accounted for by considering an equivalent true countercurrent process. This is in accordance with numerous past observations. However, the shortcoming of this approach was revealed in the analysis of the behavior of the three-section flow scheme. It is shown that average experimental concentrations of the raffinate product were lower than predicted values. However, it is important to recognize that, except for the concentration of the raffinate stream, the behavior of the three-section scheme was consistent with theoretical predictions.

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Notation

a = cross-sectional area of an adsorption column, cm^2
D = eluent flow rate, cm^3/min
D_L = axial dispersion coefficient, cm^2/min
E = extract flow rate, cm^3/min
F = feed flow rate, cm^3/min
Fr = fructose
k = overall mass-transfer coefficient, min^{-1}
K = equilibrium constant
L = length of an adsorption column, cm
R = raffinate flow rate, cm^3/min
Ra = raffinose
S = hypothetical solid recirculation rate, cm^3/min
$T6$ = dextran (M.W. $\approx$ 6,000)
$T9$ = dextran (M.W. $\approx$ 9,400)
u = hypothetical solid velocity, cm/min
v = hypothetical liquid interstitial velocity, cm/min

Greek letter

ϵ = bed voidage

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