Study on a pseudo-simulated moving bed with solvent gradient for ternary separations

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ABSTRACT

There is a trapping effect on the solute in the simulated moving bed (SMB) with solvent gradient. As the eluotropic strength of the liquid in zone II (between the extract-port and feed-port) is higher than that in zone III (between the feed-port and the raffinate-port), the solute can move forward in zone II but backward in zone III to be trapped in the two zones consequently. On this basis, a pseudo-SMB was proposed to separate the medium retained solute from a ternary mixture by selectively trapping the medium retained solute. Once the columns in zones II and III are saturated with the target solute, the solvent dissolving the feed is introduced at the feed-port to remove the least retained solute from the raffinate-port and the most retained solute from the extract-port. Finally, the target solute is recovered. A model simulation was used to investigate the behavior and performance of a system operating with this technology. The results showed that increasing the columns in zone III could trap more target solutes since the eluotropic strength decreased from zone II to zone III. Partial recovery in combination with incomplete purification could improve the separation performance in terms of the productivity and solvent consumption without losing purity.

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1. Introduction

The simulated moving bed (SMB) [1,2] consists of many chromatographic columns connected head to tail. Four ports of the feed, raffinate, desorbent and extract, dividing the SMB into four zones, are periodically switched in the liquid flowing direction to give the "simulated" counter-current movement of the solid against the liquid. As a result, the less retained solute moves forward with the liquid while the more retained solute moves backward with the solid. Thus, a binary mixture can be continuously separated into two fractions flowing out of the raffinate-port and the extract-port, respectively.

In order to enhance the separation performance, some modifications on the SMB were proposed [3–10]. For example, a gradient of temperature [5,6], pressure [7] or solvent strength [8–10] is created to make the eluotropic strength decreasing from zone I to zone IV, so that the feeding rate can be increased, and the solvent consumption can be reduced at the same time.

Recently, we found an interesting phenomenon in a solvent-gradient SMB separation process. The solutes eluted neither from the raffinate-port nor from the extract-port in 50 switches at all. After analyzing the outlet concentration of each column, all solutes were found to accumulate in zones II and III, suggesting the existence of an effect to trap the solute, which may as well be named as the trapping effect. The main reason is that the eluotropic strength of the liquid in zone II is higher than that in zone III, making the solute moving in zone II quicker than in zone III during each switch interval. As a result, the solute moves forward in zone II but backward in zone III due to the column switching. If the "simulated" counter-current movement of the solid against the liquid does not exist, the width of a peak or band will be narrowed as the upstream solute moves quicker than the downstream solute. Thereby, the solute is concentrated in a very small section of the column, which is called the focusing in the gradient chromatography [11,12] or the thermal adsorption [13,14]. However, the focusing has no selectivity, and thus it is difficult to merely focus the desired solute.

Based on the aforementioned trapping effect in SMB with solvent gradient, a novel pseudo-SMB was proposed to separate the medium retained solute from a ternary mixture [15]. As shown in Fig. 1, the solvent strength of D1 is higher than that of D2 so that the eluotropic strength decreases from zone II to zone III. Under suitable conditions, the least retained solute A moves forward and the most retained solute C moves backward in zones II and III, whereas the medium retained solute B moves forward in zone II but backward in zone III to be trapped in the two zones consequently. Unlike the focusing effect in gradient chromatography, the trapping effect in solvent-gradient SMB can selectively trap the desired solute by the counter-current movement of the solid against the liquid. Once the columns in zones II and III are saturated with solute B, the

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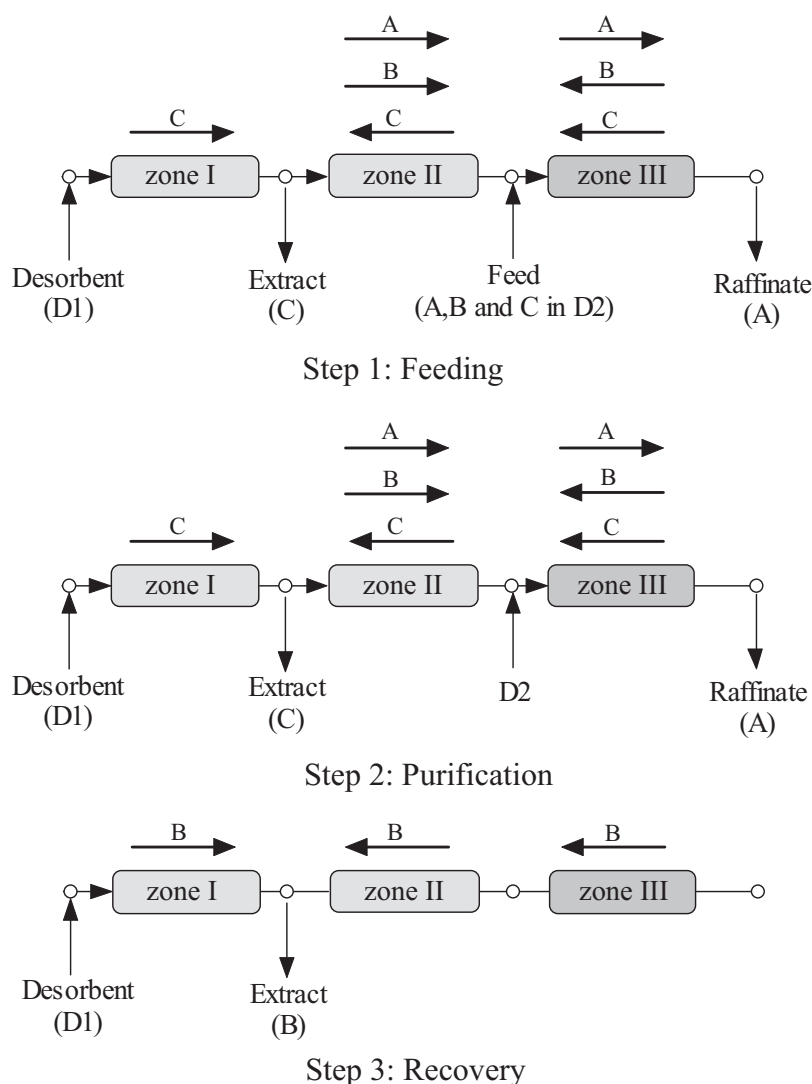 *E-mail address:* weifeng@nit.net.cn (F. Wei).

Fig. 1. Scheme of the pseudo-SMB with solvent gradient for ternary separations.

solvent dissolving the feed is introduced at the feed-port to remove solute *A* from the raffinate-port and solute *C* from the extract-port. Finally, solute *B* is recovered from the extract-port by stopping the liquid flow in zone II.

Continuous and semi-continuous multi-column processes for ternary separations are an important research field. Several new processes have been proposed for this purpose over the last years. First, a five-zone SMB was adopted to collect the medium retained solute from a side stream [16–19]. The idea could also be realized in a four-zone SMB to collect the medium and the most retained solutes from the extract-port orderly [20–22]. Second, a cascade of two SMBs in series, which was either separated [23–25] or combined in a single device [26,27], could also produce the medium retained component. The third modification was a continuous, counter-current multi-column chromatographic process incorporating modifier gradients (MCSPG process) [28,29]. The last modification of JO-SMB [25,30–33] was developed and commercialized by Organo Corporation. These schemes could produce the medium retained solute with a high purity under optimal operating conditions.

The pseudo-SMB shown in Fig. 1 is completely different from the first three modifications, but is slightly similar to the last modification. In JO-SMB, two operating modes alternate. Firstly, the feed is added in a batch chromatography mode. And then the system is operated in a SMB mode but without feeding to remove the least and most retained solutes. The medium retained solute remains motionless in zones II and III as its movement with the liquid is counterbalanced exactly by the "simulated" counter-current movement with the solid. Thus, the medium retained solute can be recovered in the next feeding duration. Nonetheless, in the pseudo-SMB in Fig. 1, the medium retained solute is trapped and consequently accumulates in zones II and III.

The pseudo-SMB based on the trapping effect provides a new idea for ternary separations by the SMB. The previous work [15] has confirmed its feasibility. However, the trapping effect was not investigated in detail to give the separation behaviors. This work will study the separation process with a model, emphasizing the evolution of the concentration bands, so as to optimize the separation process.

2. Description of SMB model

A SMB model [34–36] with consideration of the periodic switches of four ports is adopted. During each switch interval, the change of component *i* in the *j*th column numbered from the first

column in zone I is described by the following transport-dispersive equations [37].

$$\frac{\partial C_{i,j}}{\partial t} + \frac{1-\varepsilon}{\varepsilon}\frac{\partial q_{i,j}}{\partial t} = -u_j\frac{\partial C_{i,j}}{\partial Z} + D_L\frac{\partial^2 C_{i,j}}{\partial Z^2} \qquad (1)$$

$$\frac{\partial q_{i,j}}{\partial t} = k_{f,i}(q_{i,j}^* - q_{i,j}) \qquad (2)$$

where q^* is the solid concentration in equilibrium with the liquid concentration C, k_f is the lumped mass transfer coefficient, D_L is the axial dispersion coefficient, and ε is the colum voidage. The liquid flowing velocity, u, is constant under the assumption of negligible volume effects upon mixing. The Langmuir adsorption isotherm is used:

$$q_{i,j} = \frac{H_{i,j}C_{i,j}}{1 + K_A C_{A,j} + K_B C_{B,j} + K_C C_{C,j}} \qquad (3)$$

where H_i and K_i are the Henry's constant and the adsorption equilibrium constant, respectively. The dependence of H_i and K_i on the liquid composition in column j is described through the following equations [9]:

$$H_{i,j} = \frac{H_{0,i}}{(1 - X_j \cdot h_i)^{n_{0,i}}} \qquad (4)$$

$$K_{i,j} = k_{0,i}\exp\left(\frac{X_j}{a_i}\right) \qquad (5)$$

The liquid composition, X_j, denoting the volume fraction of the weak solvent with elution strength smaller than the other strong solvent, is assumed to be independent on the adsorption of the solvent. Accordingly, the change of X is merely dependent on the convection and axial dispersion as follows:

$$\frac{\partial X_j}{\partial t} = -u_j\frac{\partial X_j}{\partial Z} + D_L\frac{\partial^2 X_j}{\partial Z^2} \qquad (6)$$

At the inlet of the first column in zone I,

$$C_{i,1}(t, 0) = 0 \qquad (7)$$

and

$$X_1(t, 0) = X_{D1} \qquad (8)$$

At the inlet of the first column in zone III

$$C_{i,j}(t, 0) = \frac{C_{\text{Feed}}\cdot F_{\text{Feed}} + C_{i,j-1}(t, L)\cdot F_{\text{II}}}{F_{\text{III}}} \qquad (9)$$

and

$$X_j(t, 0) = \frac{X_{D2}\cdot F_{\text{Feed}} + X_{j-1}(t, L)\cdot F_{\text{II}}}{F_{\text{III}}} \qquad (10)$$

For the other columns,

$$C_{i,j}(t, 0) = C_{i,j-1}(t, L) \qquad (11)$$

and

$$X_j(t, 0) = X_{j-1}(t, L) \qquad (12)$$

According to the separation scheme shown in Fig. 1, besides C_{Feed} in the purification duration, F_{II} and F_{III} in the recovery duration are also zero.

After each switch interval Δt, the desorbent, extract, feed, raffinate ports are moved by one column in the liquid flowing direction. So the profiles of solute concentration and liquid composition in the $(j+1)$th column at the end of the $(n-1)$th switch interval are identical to the initial conditions in the jth column at the beginning of the nth switch interval,

$$C_{i,j}(t_n = 0, Z) = C_{i,j+1}(t_{n-1} = \Delta t, Z), \qquad (13)$$

$$q_{i,j}(t_n = 0, Z) = q_{i,j+1}(t_{n-1} = \Delta t, Z), \qquad (14)$$

Table 1
Parameters of adsorption isotherm.

Solutes	H_0	h	n_0	k_0	a
A	0.26	1.17	5.33	2.96×10^{-5}	0.0493
B	0.32	1.13	5.80	1.48×10^{-5}	0.0438
C	0.38	1.09	6.27	0.48×10^{-5}	0.0383

Table 2
Model parameters.

Parameters	Value
ε	0.70
L	10 cm
d	1.0 cm
$k_{f,A}$ ($=k_{f,B}=k_{f,C}$)	10.0 min^{-1}
D_L	0.06 cm^2/min

$$X_j(t_n = 0, Z) = X_{j+1}(t_{n-1} = \Delta t, Z), \qquad (15)$$

where t_n represents the time during the nth switch interval Δt.

For the first switch interval, the initial conditions are

$$C_{i,j}(t_1 = 0, Z) = q_{i,j}(t_1 = 0, Z) = 0 \qquad (16)$$

$$X_j(t_1 = 0, Z) = X_{\text{Feed}} \qquad (17)$$

The above model is discrete to ordinary differential equations with the method of lines [38]. The convective terms are approximated using five-point biased upwind finite difference schemes, while the dispersion items are estimated with five-point centered schemes. The ordinary differential equations are solved by the ODE15s solvers in Matlab, giving the concentration evolutions at the raffiante- and extract-port.

The detailed separation performances such as the recovery (R), the productivity (P) and the solvent consumption (S) are calculated as follows:

$$R = \frac{C_{B,R}\cdot F_{D1,R}\cdot n_R}{C_{B,\text{Feed}}\cdot F_{\text{Feed}}\cdot n_F} \qquad (18)$$

$$P = \frac{C_{B,R}\cdot F_{D1,R}\cdot n_R}{(V_{\text{col}}\cdot N_{\text{col}})\cdot(n_F + n_P + n_R)} \qquad (19)$$

$$S = \frac{(F_{\text{Feed}} + F_{D1,F})\cdot n_F + (F_{D2,P} + F_{D1,P})\cdot n_P + F_{D1,R}\cdot n_P}{C_{B,R}\cdot F_{D1,R}\cdot n_R} \qquad (20)$$

where $C_{B,\text{Feed}}$ is the concentration of solute B in the feed stream in step 1, $C_{B,R}$ is the concentration of solute B in the extract stream collected in step 3, $F_{D1,F}$, $F_{D1,P}$ and $F_{D1,R}$ are the flow rates of solvent $D1$ in steps 1–3, F_{Feed} is the feeding rate in step 1, $F_{D2,P}$ is the flow rate of solvent $D2$ in step 2, and n_F, n_P and n_R are the number of switches in steps 1–3.

3. Parameters of model simulation

The dependence of isotherms on the liquid composition must be available in advance. This is a very difficult task especially for nonlinear adsorption. We adopt the work of Morbidelli's group [9]. As listed in Table 1, solutes A and B are (S)-(+)-ionone and (R)-(−)-ionone, whereas solute C is an imaginary component eluted after (R)-(−)-ionone. From the dependence of H on X shown in Fig. 2, the solutes are adsorbed more strongly as the content of the weak solvent in the mobile phase increases. Since selectivity is given by the ratio of H_A/H_B or H_B/H_C, it is also a function of the liquid composition and varies in the range of 1.29–1.42. On the other hand, the nonlinear adsorption behavior becomes more distinct with increasing X as shown in Fig. 3.

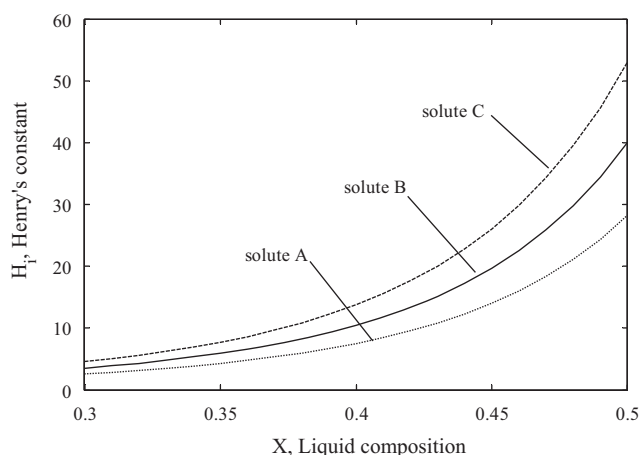The other model parameters are listed in Table 2.

Fig. 2. The effect of the liquid composition, X, on the Henry's constants of solutes A, B and C.

4. Results and discussions

4.1. Designing the separating conditions

In the design procedure for binary separations with conventional SMB, equilibrium theory incorporated with true moving bed model is often adopted to give the initial separating conditions, i.e., Triangle Theory gives a separation region in ideal cases [39,40]. Nevertheless, the initial separating conditions are only possible and rough, not necessarily feasible when taking into account the non-ideal cases. These initial conditions must be screened and tuned with rigorous models or experiments to give the feasible separations using a trial and error procedure.

In comparison with the conventional SMB, the pseudo-SMB for ternary mixture is more complex, making the design very difficult. We have developed a designing method for the linear adsorptions. [15] If the deviation from linear adsorption is not very significant, the separating conditions derived from the method can applied to nonlinear adsorptions. Thus, the feed concentration was set to 1 mg/mL for each solute. The values of X_{D1} and X_{D2} were set to 0.3 and 0.5, respectively. The initial separating conditions were first obtained, which was a separation region in the plane of $F_{II}\Delta t$ and $F_{III}\Delta t$ as shown in Fig. 4. Then, model simulations were used to screen the initial conditions, giving the feasible operating conditions (see Table 3) in non-ideal and nonlinear adsorptions.

4.2. The effect of column configuration

Intuitively, increasing the columns in zone III should be able to trap more solute B because the eluotropic strength decreased from zone II to zone III. As listed in Table 4, the columns in zones II and III were changed without varying the total columns in the SMB. The feeding duration for each configuration lasted for 40 switches. The time of solute B leaking out of zone III, called as the leakage point, was used to assess the different column configuration. It was found that the leakage point delayed with increasing the columns in zone III. Thereby, the amount of solute B leaking out of zone III decreased, and the trapped amount of solute B increased consequently. For the configuration of 1/1/6 (one column in zone I, one column in zone II, and six columns in zone III), a small portion of solute B had leaked from the extract-port as there was only one column in zone II. However, the amount of solute B to be trapped was the largest, so the configuration of 1/1/6 should be adopted.

Besides, increasing the columns in zone III could reduce the purification duration for the complete removal of the impurities, indicating solute C had a higher simulated counter-current movement velocity in zone III than in zone II, or solute A had a higher moving velocity in zone III than in zone II under the separating conditions. However, the conclusion is not unconditional as the movement of solutes is dependent on the adsorption isotherm and the operating conditions. For example, if the eluotropic strength of the liquid $D2$ is increased, making the counter-current movement of solute C in zone III slower than in zone II, the complete purification duration will increase with enhancing the columns in zone III.

4.3. The effect of the feeding duration

For the column configuration of 1/1/6, the B's leakage point was 34 switches. However, the actual feeding duration should not be larger than 34 switches. As shown in Table 5, the optimum feeding duration was 28 switches. In this case, the amount of solute B to be trapped was the most, and in the mean time, the complete purification duration was relatively short.
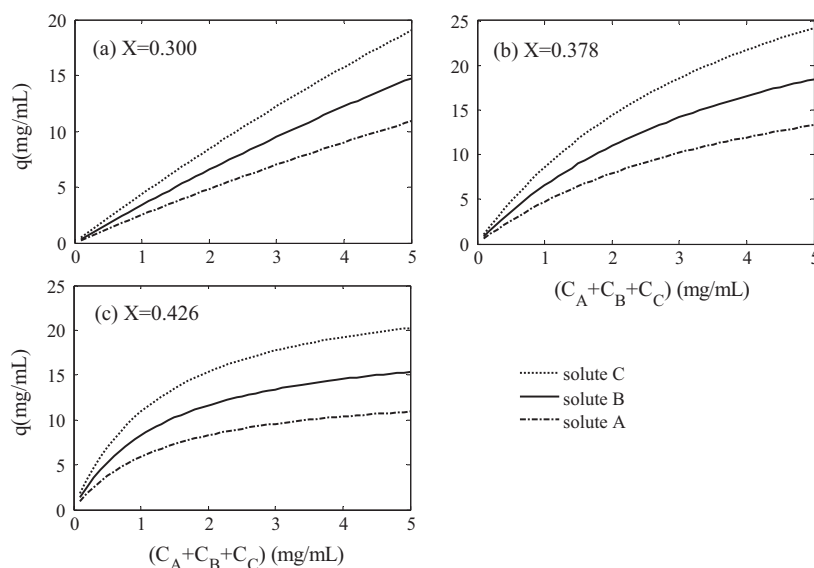

Fig. 3. Adsorption isotherms at different liquid compositions; ratio $C_A/C_B/C_C = 1/1/1$.

Table 3
Operating conditions for pseudo-SMB.

	X_{D1}	X_{D2}	Δt (min)	F_I (mL/min)	F_{II} (mL/min)	F_{III} (mL/min)	$C_{Feed,i}$ (g/L)
Step 1	0.3	0.5	5	4.50	2.95	4.85	1.0
Step 2	0.3	0.5	5	4.50	2.95	4.85	0
Step 3	0.3	–	5	4.50	0	0	–

Table 4
The effect of column configuration on the separation.

Column configuration	1/4/3	1/3/4	1/2/5	1/1/6
Feeding duration (switches)	40	40	40	40
Purification duration for completely removing impurities (switches)	125	105	80	39
B's leakage point (switches)	16	21	27	34
Amount of B leaking from the raffinate-port (mg)	310.0	264.7	215.7	140.6
Amount of B leaking from the extract-port (mg)	0	0	0.1	18.6
Amount of B to be trapped (mg)	70.0	115.3	164.2	220.8

Table 5
The effect of feeding duration on the separation.

Feeding duration	24	26	28	30	32	34
Complete purification duration	54	44	40	40	40	40
Amount of B feeding to SMB (mg)	228	247	266	285	304	323
Amount of B leaking out of the raffinate-port (mg)	0.1	1.3	13.5	34.1	55.4	76.7
Amount of B leaking out of the extract-port (mg)	25.0	20.3	18.5	18.6	18.7	18.8
Amount of B to be trapped (mg)	202.9	225.4	234	232.3	229.9	227.5

The amount of solute B leaking out of the raffinate-port increased with the feeding duration. In contrast, the leakage out of the extract-port decreased rapidly from 25 mg to 18.5 mg, and then increased slowly to 18.8 mg. The result was reasonable. When the feeding duration was less than 28 switches, the trapped B's amount increased with the feeding duration, thus increasing its moving velocity in zone II and reducing the leakage out of the extract-port consequently. On the other hand, when the feeding duration was larger than 28 switches, the B's moving velocity in zone II had been relatively stable, but the trapped B's amount still increased to enhance the height of B's band in zone II, which slowly increased the leakage out of the extract-port.
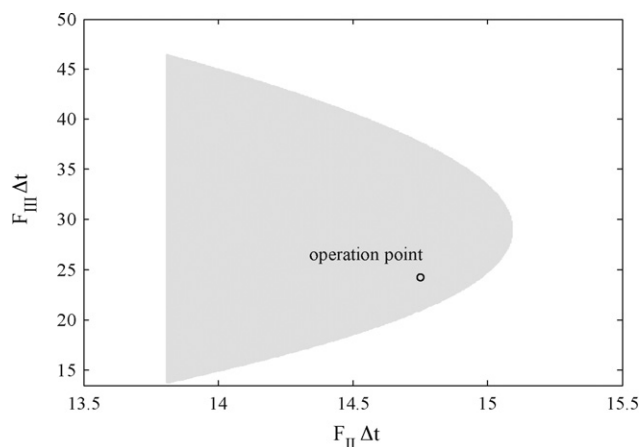
4.4. The evolution of concentration bands

As shown in Fig. 5, in the feeding duration of switches 1–28, solutes A and C eluted out of the raffinate- and extract-port respectively, whereas solute B was accumulated in zones II and III. In the purification duration of switches 29–68, the solvent dissolving the feed, instead of the feed solution, was introduced at the feed-port. Solutes A and C continued to be removed to purify solute B. The

**Fig. 4.** Separation region in the plane of $F_{II}\Delta t$ and $F_{III}\Delta t$.

elution curves in Fig. 6 showed that a small portion of solute B had leaked out of the raffinate-port in switches 34–50. After the complete removal of impurities, solute B was recovered from the extract-port in switches 69–80 by stopping the liquid to flow in zones II and III. Actually, the complete recovery took just 7 switches, as only seven columns in zones II and III were demanded to move by zone I successively to recover solute B.

From the concentration bands shown in Fig. 5, the feature of nonlinear adsorption was very distinct even at the low feed concentration of 1 mg/mL. For example, there were two plateaus on the concentration band of solute A at the end of switch 28. One located in column 8 was formed by the well-known displacement effect of solute B, and the other located in column 5 was formed by the displacement of solute C. The plateau on the concentration band of solute B was also caused by the displacement of solute C. The competitive and nonlinear adsorption of solutes was the main reason causing the displacement effect.

In addition, there were two plateaus on the band of solute B in the purification duration as shown in Fig. 5b–e. They were formed because there was a dilution effect at the feed-port caused by the introduction of solvent D2, the plateau at the back of the band would immediately be split into two new plateaus at the beginning of a switch interval. The new plateau before the feed-port continued to move forward and reached the front of the band at the end of the switch interval, which would be smoothed down in the next switch interval. On the other hand, as the eluotropic strength of the liquid in the column right after the feed-port increased gradually in a switch interval, the amount of solute B desorbed from the solid would also increase, causing the other new plateau after the feed-port to become broad. The new broad plateau would be split into two parts again in the next switch interval. If the dilution effect was eliminated by stopping the solvent D2 to flow into the feed-port, the new plateau after the feed-port should become high, not broad.

Fig. 7 gave the profiles of the liquid composition at different time in a switch interval of the feeding or purification duration. There were two steps of compositions with values of 0.3 and 0.378, respectively. On the latter step, there always was at least one plateau of 0.426. The continuous tail of the plateau was formed about at the 2nd minute in the present switch interval, and then moved out of zone III at the end of the next switch interval. Thus,

(a) switch 28

(b) switch 40

(c) switch 50

(d) switch 60

(e) switch 68
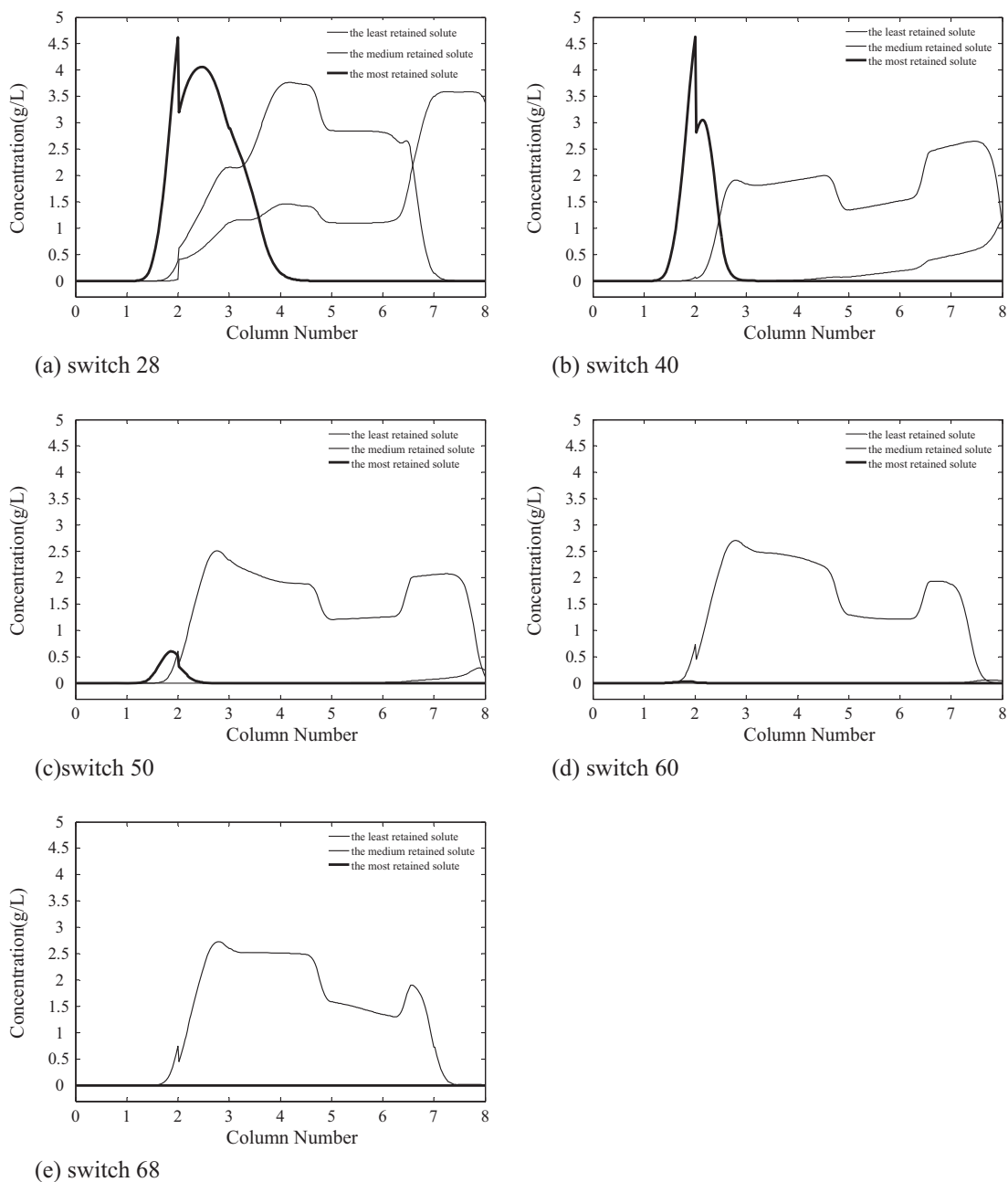
Fig. 5. Concentration bands in the pseudo-SMB with a column configuration of 1/1/6 at the end of switches 28, 40, 50, 60, and 68 (a to e).

the continuous tail took about 8 min to move out of zone III, during which the tail moved over 7 columns. In 8 min, the volume of liquid flowing out of zone III was 38.8 mL, very close to the dead volume of seven columns (38.5 mL). The result confirmed the accuracy of the solution of the model with the method of lines.

### 4.5. Incomplete purification and partial recovery

Looking at Figs. 5 and 6, the amount of impurities remaining in zones II and III had been very small since switch 50, indicating that the purification could be stopped early without losing the purity. As listed in Table 6, when the purification duration reduced from 40 to 22 switches, the purity was decreased slowly from 99.88% to 96.30%. On the other hand, the separation performances in terms of the recovery, productivity, and solvent consumption were all

improved greatly. Accordingly, the purification duration should be shortened as possible if the demand on the purity was not rigorous.

Moreover, in the purification duration, solute A always moves forward to the raffinate-port, whereas solute C moves backward to the extract-port. Thus, in the late purification duration, e.g., switches 60–68, solute A was remained in the last column in zone III, whereas solute C was remained in the first column in zone II as shown in Fig. 5. As a result, when the purification step was stopped early to recover solute B by shifting all the columns in zones II and III by zone I successively, the eluant from the first column in zone II and the last column in zone III could be discarded to improve the purity. For example, for the purification duration of only 27 switches, the purity was enhanced from 98.81% to 99.21% with a small reduction in the recovery if the eluant from columns 3–8, rather than from columns 2–8, was collected as the product stream. Discarding the eluant from column 8 could also improve the purity; nevertheless,

Table 6
The effect of the purification duration and partial recovery.

Feeding duration (switches)		28	28	28	28
Purification duration (switches)		22	27	32	40
Total recovery (columns 2–8)	Purity (%)	96.30	98.81	99.58	99.88
	Recovery (%)	89.91	89.07	88.20	87.02
	Productivity (g L^{-1} h^{-1})	0.802	0.730	0.669	0.590
	Solvent consumption (L/g)	7.35	8.09	8.86	10.08
Partial recovery (columns 3–8)	Purity (%)	97.90	99.21	99.66	99.89
	Recovery (%)	89.50	88.57	87.68	86.50
	Productivity (g L^{-1} h^{-1})	0.798	0.726	0.665	0.586
	Solvent consumption (L/g)	7.38	8.14	8.91	10.14
Partial recovery (columns 2–7)	Purity (%)	97.01	99.33	99.86	99.99
	Recovery (%)	77.37	80.10	82.98	86.11
	Productivity (g L^{-1} h^{-1})	0.690	0.657	0.630	0.584
	Solvent consumption (L/g)	8.54	9.00	9.41	10.19
Partial recovery (columns 3–7)	Purity (%)	98.90	99.78	99.96	99.99
	Recovery (%)	76.96	79.60	82.46	85.59
	Productivity (g L^{-1} h^{-1})	0.686	0.653	0.626	0.580
	Solvent consumption (L/g)	8.59	9.06	9.47	10.25
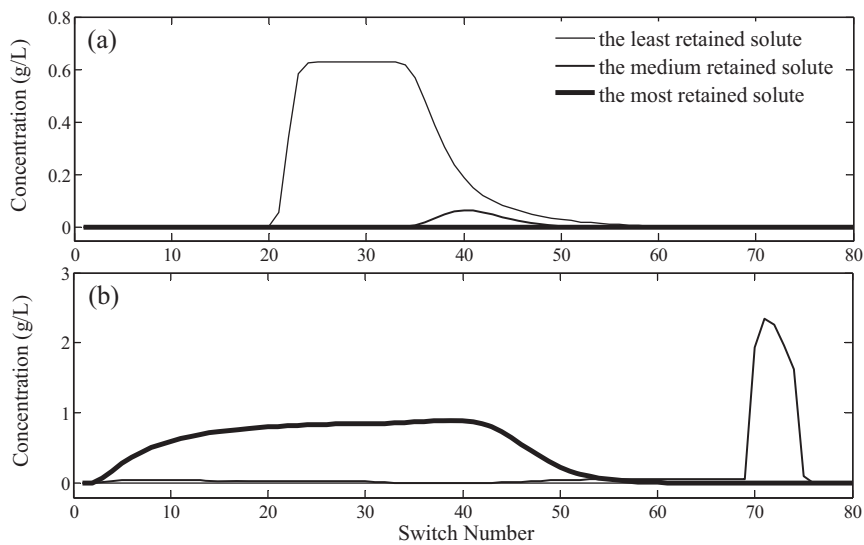
Fig. 6. Elution curves at the raffinate-port (a) and the extract-port (b) in the pseudo-SMB with a column configuration of 1/1/6.
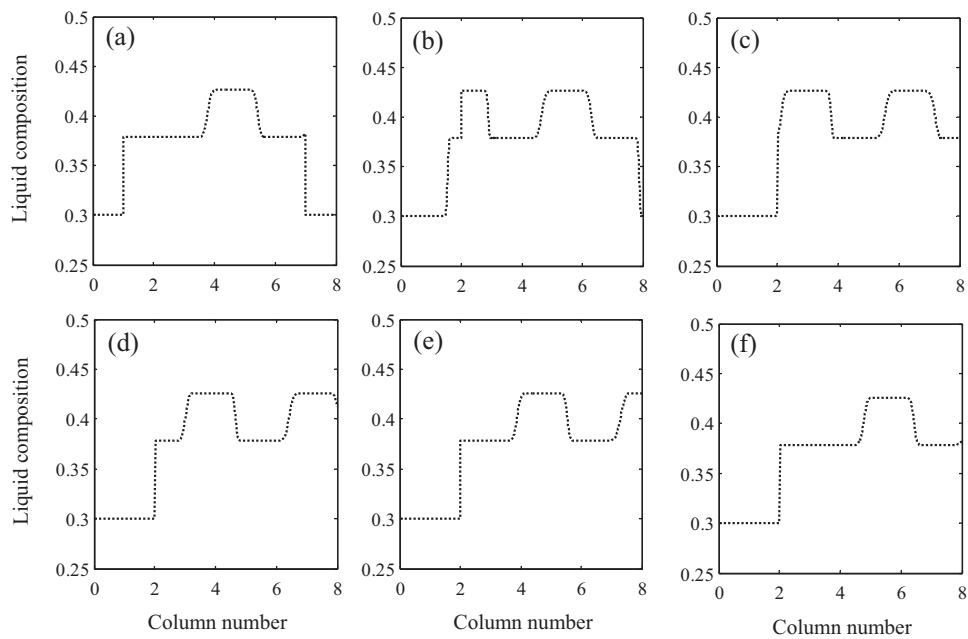
Fig. 7. The profiles of the liquid composition at 0, 1.0, 2.0, 3.0, 4.0 and 5.0 min in a switch interval (a–f), respectively.

the reduction in the recovery was relatively large, e.g., from 89.07% to 80.10%.

5. Conclusions

A pseudo SMB for separating the medium retained solute from a ternary mixture consisting of the least, medium and most retained solutes (*A–C*) was investigated. The separation process included three steps. In the first feeding step, solute *B* was trapped and accumulated in zones II and III while solutes *A* and *C* were removed from the raffinate and extract ports, respectively. In the second purification step, the solvent dissolving the feed was introduced at the feed port to remove the remaining solute *A* from the raffinate-port and the remaining solute *C* from the extract-port, so solute *B* trapped in zones II and III was purified. Finally, solute *B* was recovered from the extract port by stopping the liquid flow in zone II.

A model investigation on a system operating with this technology was conducted to reach the following conclusions.

(1) The medium retained solute was trapped in zones II and III to be saturated eventually and thus would leak out of zone III. As the adsorption affinity increased with reducing the eluotropic strength, the amount of solute *B* to be trapped increased with increasing columns in zone III.
(2) Solutes *A* and *C* could be removed completely by purification, but the purification duration will be very long. Incomplete purification in combination with partial recovery could shorten the purification without losing purity, and meanwhile improved the separation performance in terms of the productivity and solvent consumption.

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