Experimental Evaluation of Designs for the Simulated Countercurrent Moving Bed Separator

Anna Lee Y. Tonkovich and Robert W. Carr
Dept. of Chemical Engineering and Materials Science, University of Minnesota, Minneapolis, MN 55455

Simulated countercurrent chromatography is a continuous flow method for separation of binary mixtures or for separation of multicomponent mixtures into two fractions. Countercurrent flow is simulated by moving the feed point past several fixed adsorbent beds at a rate between the single column breakthrough time of each component. The number of columns and the location of the inlet and outlet port were varied to investigate product purity and productivity. Concentration profiles of the effluent product streams were measured. The maximum product concentration exceeds the feed concentration during part of the feed-switching cycle and drops to zero during part of the cycle. Three configurations were tested with the total number of columns varying between three and eight. For the test separation chosen, gaseous propylene and dimethyl ether on Chromosorb 101, high purity (> 99%) product streams were obtained with an optimal four-column configuration using three desorbent streams.

Introduction

Simulated countercurrent flow is an engineering innovation to improve the efficiency and throughput of separations based on adsorption. Unlike traditional pulsed input chromatography, simulated countercurrent technology permits the continuous separation of large volumes while minimizing adsorbent usage.

Countercurrent flow is simulated to avoid flow channeling, and particle attrition and abrasion, which are often encountered with moving-solids streams. Simulated countercurrent flow is based on the concept that separation can be achieved by moving the feed point past fixed-bed solid adsorbent sections, rather than flowing the solid past a fixed-feed point as in true countercurrent flow. In simulated countercurrency the more weakly sorbed component(s) move ahead of the feed point, and the more strongly sorbed component(s) lag behind it. In a previous article, the design and performance of a simulated countercurrent moving-bed chromatographic separation (SCMCS) having four adsorbent beds was reported (Fish et al., 1993). In tests of the separation of propylene from dimethyl ether, for which the separation factor is 2, it was found that the product stream containing propylene was always contaminated with a few percent of dimethyl ether due primarily to heterogeneity of the adsorbent surface. In the present work, we have investigated alternative configurations of the SCMCS that give improved separations. The gas–solid system of this study contrasts most previously reported work, which has been on liquid–solid systems.

Background

The Hypersorption process, developed in 1947, was the first commercial continuous countercurrent adsorption operation (Freund et al., 1957). A feed mixture of light and heavy gases entered at the center of a single large column (approximately 85 ft long and 4 1/2 ft in diameter) into a slowly downward moving stream of activated carbon and an upward flow of a carrier gas. The countercurrent flow of the two phases acted to separate the solutes more efficiently than a fixed-bed adsorber. Flow problems were found with this process, including channeling of the gaseous stream, and abrasion and attrition of the moving-solid particles.

The response to the problems associated with the moving solids stream has been to simulate countercurrent flow. The Sorbex process developed by UOP is one such example (Broughton, 1968, 1984; Broughton et al., 1970; De Rosset et al., 1981). Several large-scale applications called Parex, Olex, Molex, Sarex, Cresex, and Cymex are all variations of the basic Sorbex idea in that they are processes that apply simu-

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Correspondence concerning this article should be addressed to R. W. Carr.
Current address of A. L. Y. Tonkovich: Pacific Northwest Laboratory, Richland, WA 99352.

lated moving-bed technology to facilitate binary liquid phase separations.

Three different types of simulated countercurrent experimental designs for simulated countercurrency have been explored. The first is a semicontinuous moving feed injection system, where the feed port is moved once past a series of inlet points in a discretized manner. The second is a small-scale system that uses a single desorbent stream, and the third employs several carrier streams. The use of multiple desorbent streams allows for improved product purity at the expense of increased carrier use.

Semicontinuous moving feed systems

Several examples of the moving feed point injection system have been presented (Ha et al., 1987a,b; Hidajat et al., 1986; McGary and Wankat, 1983; Wankat, 1977, 1984; Wankat and Ortiz, 1982). The feed point either moves once along a single fixed column or once past several columns connected in series. The liquid desorbent or carrier gas is continuously fed at the bottom of the column. The injection point is moved at a rate between the convective elution velocity of the two solutes. This process allows for a greater throughput of feed over that obtained in preparative chromatography. The column volume is used more efficiently, but in a semicontinuous manner.

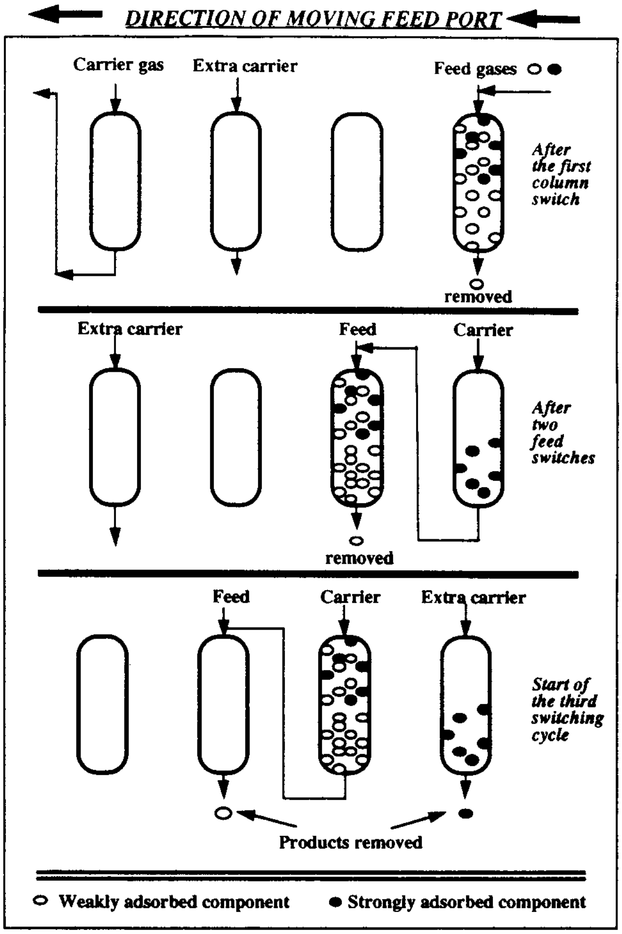


Figure 1. Achieving simulated countercurrent flow.

Each port is moved one column to the left at each switching time.

Single-desorbent simulated-countercurrent processes

Small-scale continuous simulated countercurrent processes have been described and are operated in a similar fashion to the Sorbex separators (Broughton, 1984; Ching et al., 1985c; De Rosset et al., 1981; Huang et al., 1986; Kubota et al., 1989; Ruthven, 1984; Ruthven and Ching, 1989; Saska et al., 1992; Storti et al., 1992). Either a single long column with multiple feed introduction ports, or multiple columns and multiple feed ports are used in these apparatuses. In all cases, the columns are grouped into four operating zones (for product removal and adsorbent regeneration) with the numbers of columns per zone ranging between one and four. A single carrier stream is split within the system and used to separate and remove the two components.

Multiple-desorbent simulated-countercurrent processes

This final class of separators uses multiple desorbent carrier streams. One liquid product stream is completely removed at one port, and the other product is removed with an isolated carrier stream (Barker, 1971; Barker and Critcher, 1960; Barker and Deeble, 1973; Barker and Abusabah, 1985; Barker et al., 1978, 1983, 1990; Ching and Ruthven, 1985a,b, 1986; Ching et al., 1987, 1988, 1991; Hashimoto et al., 1983a,b, 1987, 1989, 1990a,b; Kishihara et al., 1989; Maki et al., 1987; Ruthven, 1984; Ruthven and Ching, 1989; Szepesy et al., 1975). These systems are also subdivided into four zones with multiple columns (ranging between two and six) per section. There are both advantages (increased product purities) and disadvantages (increased desorbent requirements) associated with this mode of operation.

Simulation of Countercurrent Flow

In binary gas phase separations, one component must be more strongly sorbed than the other. In true countercurrent flow, the strongly sorbed component convectively travels with the solid phase, while the weakly sorbed component is swept out of the system with the gaseous or liquid phase. In simulated countercurrent flow this effect is approximated without the physical movement of the solid adsorbent.

In simulated countercurrency, the feed introduction port is moved in discrete steps along a single fixed adsorbent bed, or along a series of shorter packed columns. When the end of the bed, or of the columns, is reached the feed is returned to the beginning and the process repeated. The column switching period (the time interval that a feed enters a column before it is moved to the next column) is set between the breakthrough time of the two components. The weakly sorbed component elutes from the feed column before the feed is moved to the next column (feed switching). The strongly sorbed product is retained in the feed column and is desorbed two switches later. Countercurrent flow is simulated because the strongly sorbed component travels behind the moving-feed port, while the weakly sorbed component moves ahead of the moving-feed port. This effect is shown in Figure 1.

Simulated countercurrent flow produces concentration profiles that differ from those produced in true countercurrent separators, where constant concentration product streams are continuously removed. Periodic concentration

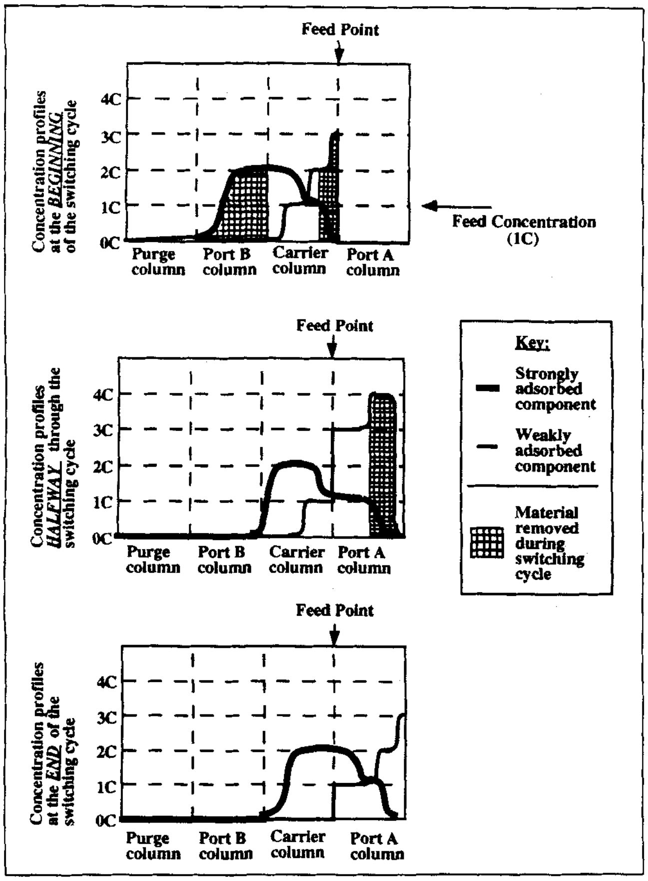


Figure 2. Column concentration profiles.

Breakthrough ratios—weakly adsorbed component = 4:3, strongly adsorbed component = 2:3.

profiles are formed during operation of simulated countercurrent separators. After the feed is switched, the solutes enter a clean (regenerated) column. A "dead-time" concentration (zero) exists before material elutes from this column. When the weakly sorbed product does elute from the feed column, it has a higher concentration than the feed. A steady-state mass balance over each cycle shows that the product gas concentration during the non-dead time must be greater than the feed-gas concentration. This effect has been explained previously (Fish et al., 1988, 1993).

The important parameter for determining the cyclic concentration profile is the breakthrough ratio [$r(i)$], where $r(i)$ is defined as the switching time divided by the breakthrough time of component i. For separation to occur, the breakthrough ratio for the weakly sorbed component must be greater than one, while that for the strongly sorbed component must be less than one.

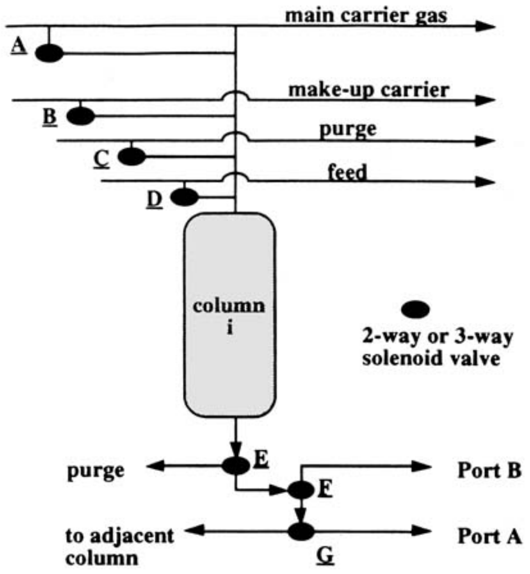
For favorable adsorption isotherms and dispersionless flow, sharply banded concentration profiles propagate through the system. Figure 2 shows the "concentrating" effect for breakthrough ratios of 4:3 and 2:3 for the weakly and strongly sorbed component, respectively. In this example, the weakly sorbed component is removed during the final quarter of each switching cycle (at port A) in a band that is four times the feed concentration. The strongly sorbed component is removed during the first half of each switching cycle (at port B) in a band that is two times the feed concentration.

Experimental

A test system of propylene and dimethyl ether in a nitrogen carrier gas was chosen for continuity with previous work (Fish et al., 1993). The retention time ratio is approximately two on the Chromosorb 101 adsorbent at room temperature. Dimethyl ether is the more strongly sorbed component.

The columns were packed with equal weights (approximately 9.5 g) of 60/80 mesh Alltech Chromosorb 101. The length of each stainless steel column was 12 in. (305 mm) with a 1/2-in. (13-mm) outside diameter and a wall thickness of 0.035 in. (0.89 mm). The ends of the columns were fitted with 1/2-in. (13-mm) to 1/8-in. (3.2-mm) Swagelock reducing unions and packed with glass wool to hold the adsorbent in place. All connections between columns were made with 1/8-in. (3.2-mm) OD stainless steel tubing.

Six solenoid valves were open at any time to allow the gases to enter and exit the appropriate columns. The inlet valves (feed, carrier, and purge) were two-way valves, and only one of each was open at a time. The others were 3-way solenoid valves, directing effluent flows to the next column or out of the system. The valve diagram for each identical column is presented in Figure 3. The exit of each column must be capable of four options: (1) to send the stream on to the next column; (2) to take the stream out of the system; (3) to take



Valve	Column number			
	1	2	3	4
A	0	0	0	1
B	0	0	1	0
C	0	1	0	0
D	1	0	0	0
E	0	1	0	0
F	0	0	1	0
G	0	0	0	1

Valve sequence for feed on column 1 (0=off,1=on)

Figure 3. Valve diagram for each identical column.

The position of each valve is shifted to the right when column 2 is the feed column, and so on.

part of the stream out and let part go on to the next column; or (4) to send the effluent to be mixed with the feed stream. The entrance of each column allowed for the introduction of feed and carrier gases as well as the stream exiting from the adjacent column.

A Varian 3700 gas chromatograph (GC) with a flame ionization detector (FID) was used to measure effluent gas concentrations. A computer-controlled sampling valve pulled a sample from the effluent stream every 40 s for analysis. The samples were analyzed by a 10-in. (254-mm), 1/8-in. (13-mm) OD GC column, also packed with Chromosorb 101. The oven temperature was 40°C, and the detector temperature was 250°C. The retention times of the feed components, propylene and dimethyl ether, were approximately 20 and 30 s, respectively, in a nitrogen carrier gas flow rate of 30 mL/min. The detector signal was routed back to the computer for analysis. The GC peak height was calibrated as a function of mole fraction.

Concentrations of propylene and dimethyl ether were measured as a function of time. The breakthrough time for propylene in each column of the separator was 42 s at a carrier flow rate of approximately 800 mL/min, and the breakthrough time of dimethyl ether was 102 s. The breakthrough curve for propylene was a sharp, S-shaped curve, while that for dimethyl ether was somewhat broader.

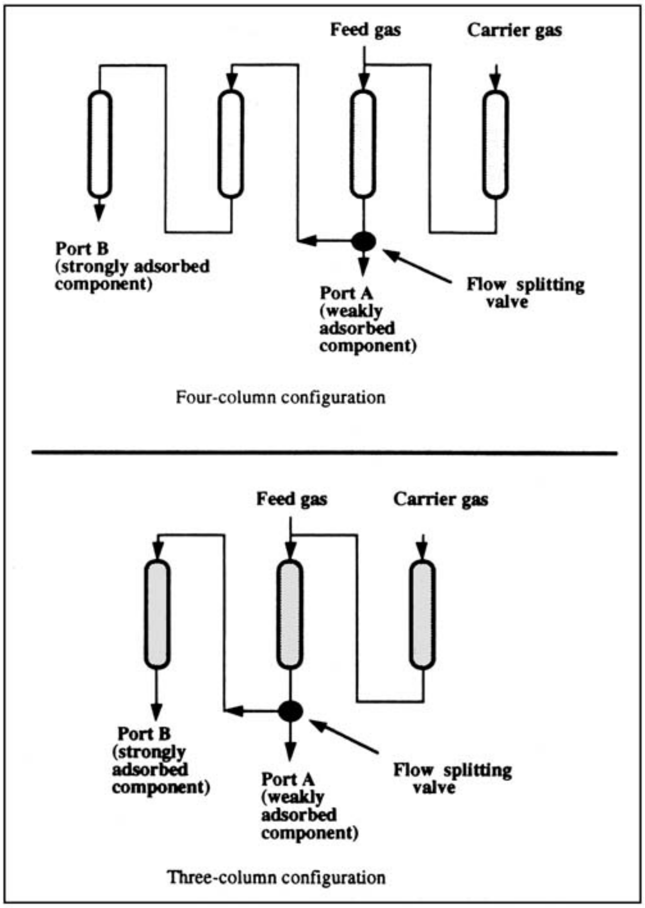


Figure 4. Single-desorbent configuration.

Four- and three-column configurations; each inlet and outlet port is moved one column to the left at every switching time.

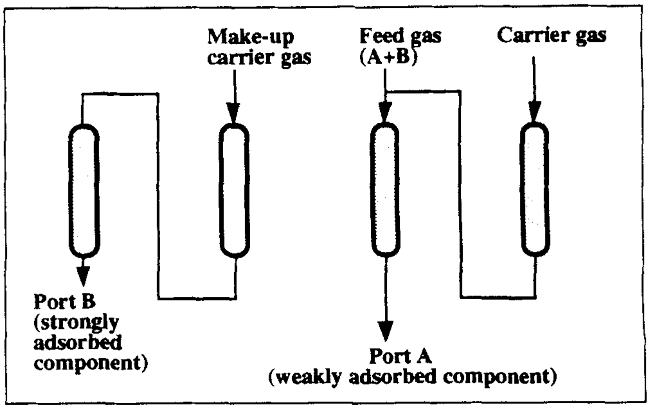


Figure 5. Two-desorbent configuration.

Four-column configuration; each inlet and outlet port is moved one column to the left at every switching time.

Experimental configurations

Three configurations (single-desorbent mode, an intermediate two-desorbent design, and a final three-desorbent separator) were evaluated and optimized. Varying numbers of columns, ranging from three to eight, were also investigated.

A design that uses one desorbent stream was investigated initially and was similar to an apparatus described previously (Fish et al., 1993). The effluent removed at the end of the feed column (port A) was split into two streams. One stream was removed from the system and analyzed. The other stream was sent ahead through the post-feed column and then on to desorb the strongly sorbed component in the port B column. Three- and four-column configurations are presented in Figure 4. The location of inlet and outlet ports are shown during one switching period; all ports move one column to the left at each switching time.

There are advantages and disadvantages to this configuration. Dead time, the time when the component concentration is equal to zero before the weakly sorbed component elutes, is reduced. Sending part of the weakly sorbed product stream to the next column saturates the post-feed column. After a column switching, the new feed column already contains some of the weakly sorbed component, thus reducing the dead time. Another advantage is the reuse of the carrier stream. If an expensive carrier gas or liquid were used, this configuration would present significant economic savings.

The use of split flow also presents disadvantages. The amount of carrier available to desorb the strongly sorbed component decreases as the split flow increases. Residual material then remains behind to decrease the purity of the weakly sorbed product stream several switches later.

The apparatus design was modified by adding an additional carrier gas to completely desorb the strongly sorbed component in the port B column. Figure 5 presents this configuration, which was explored with both three- and four-column separators.

The final design was a three-desorbent separator that added a purge stream to "clean" the post-feed column (the column that becomes the feed column at the next feed switching). The gas flowrate through this column was set much higher than both carrier flows to remove all residual material. This configuration was evaluated with both four and eight columns and is presented in Figure 6.

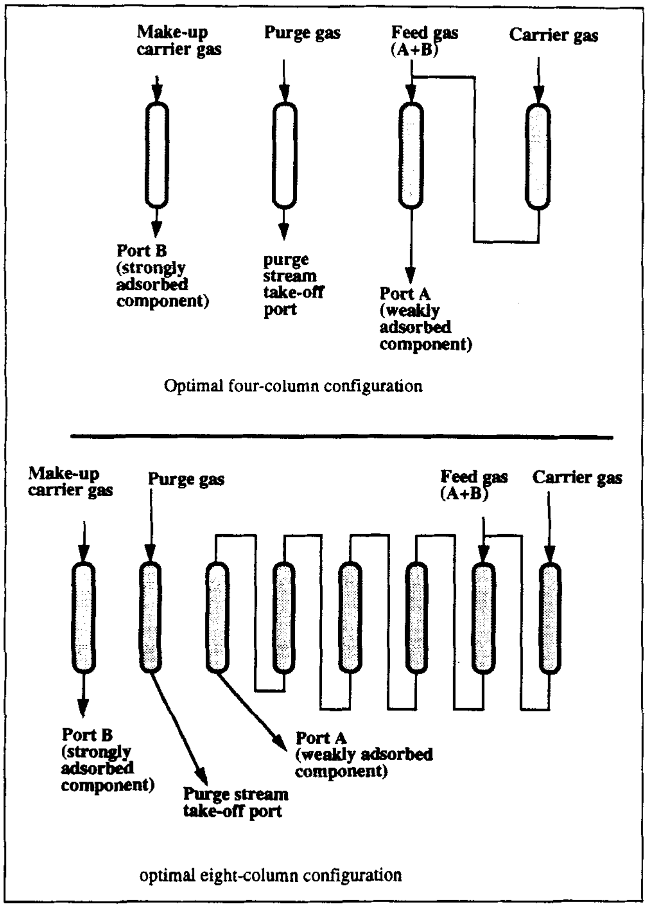


Figure 6. Optimal (three-desorbent) configuration.

Four- and eight-column design; each inlet and outlet port is moved one column to the left at every switching time.

Data Format

The data presented in Figures 7 through 10 are the measured concentrations as a function of time. Concentrations could not be measured continuously by analytical gas chromatography, and were determined every 40 s. The large open squares represent the exiting concentration of propylene (A) removed at port A (the weakly sorbed component effluent removal port) and the large open circles represent the exiting concentration of dimethyl ether at port B (the strongly sorbed component effluent removal port). In these figures the concentrations are not constant. They are periodic at steady state, and the concentration changes from zero to several times the inlet feed concentration during every switching period.

For the initial configuration, where the use of a split flow reduced the dead time, the concentrations varied between the feed concentration and three times the feed concentration. The product effluent flow rate, though, was lower than the inlet feed plus carrier flow rates. For the other configuration, the propylene (weakly sorbed solute) concentration changed from zero (immediately after a feed switching) to two or three times the feed concentration during the later stages of the switching period. The small closed squares and circles represent impurities in the product streams.

Another feature of each figure is the reported maximum concentration. It appears that the maximum is periodic as well, but this is an artifact of the measurement technique. The apparent scatter in the data is a result of sampling once every 40 s, which corresponds to different times along the breakthrough curve.

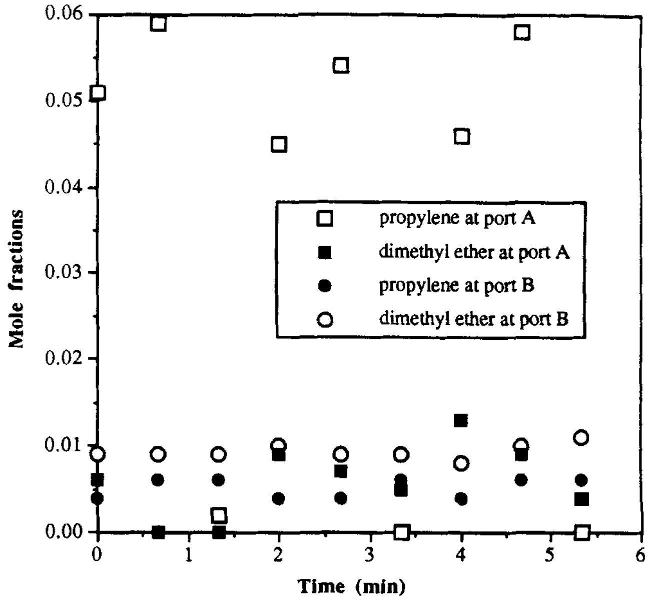


Figure 7. Single-desorbent configuration—experimental results.

Four column configuration; switching time = 60 s; $r(A)$ = 1.43 and $r(B)$ = 0.6; inlet mole fractions—propylene = 0.018 and dimethyl ether = 0.012.

Results and Discussion

The single-desorbent experimental apparatus is shown in Figure 4. The switching times were set between the break-

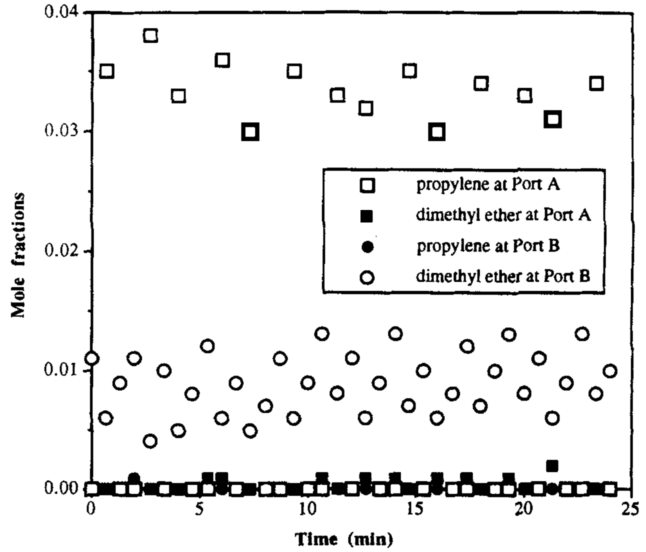


Figure 8. Optimal column configuration—experimental results.

Four-column configuration; switching time = 65 s; $r(A)$ = 1.5 and $r(B)$ = 0.7; inlet mole fractions—propylene = 0.012 and dimethyl ether = 0.012.

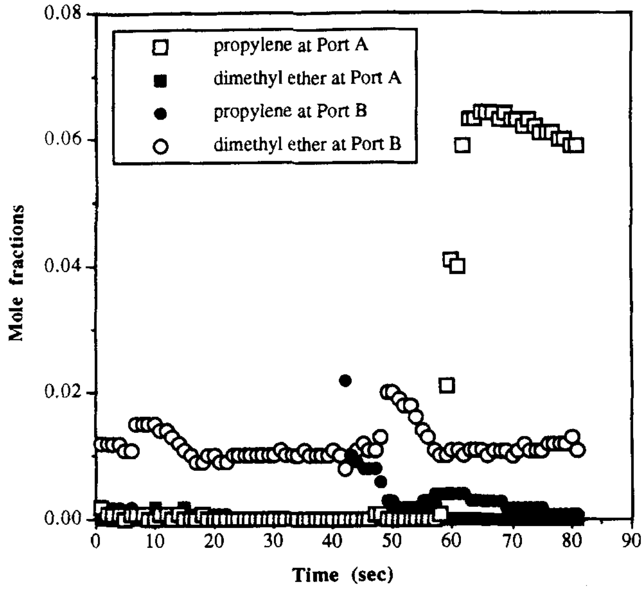


Figure 9. Optimal column configuration—experimental results.

Four-column configuration; switching time = 81 s; $r(A)$ = 1.35 and $r(B)$ = 0.63; inlet mole fractions—propylene = 0.017 and dimethyl ether = 0.017.

through times of the two components. Figure 7 presents the steady-state results at a switching time of 60 s. Steady state was achieved after many feed switches (> 100). The breakthrough ratios were 1.43 and 0.6 for propylene and dimethyl ether, respectively. The overall carrier flow rate was 800 mL/min, and each split flow rate was 400 mL/min. Feed flow rates of 15 mL/min and 10 mL/min for propylene and dimethyl ether, respectively, were used. The corresponding feed mole fractions were 0.018 and 0.012. Figure 7 shows the "concentrating" effect at port A (propylene product port), but this was not observed at port B (dimethyl ether product port) because of peak tailing. The dimethyl ether stream is dispersed, with each feed switch broadening the concentration wave, which for this component is the back of the input square wave, or the desorption curve. A high level of impurity is seen in the product streams. The propylene purity at port A, which was calculated as the average fraction of the desired product in the total port A effluent, was less than 90%.

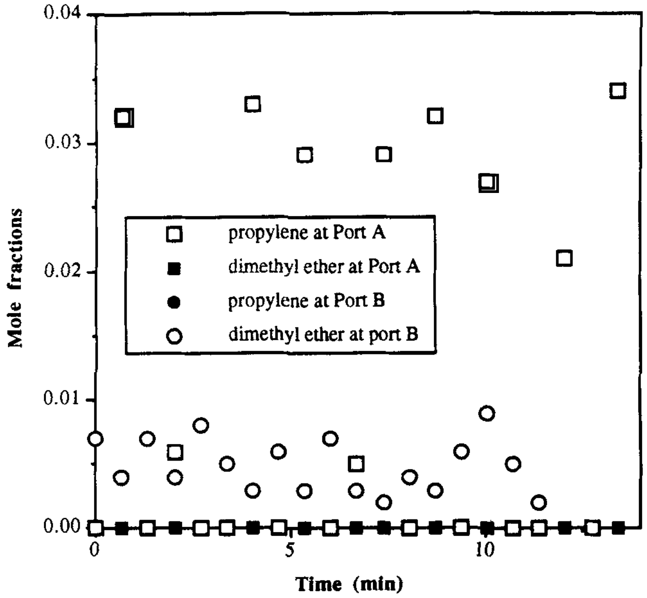


Figure 10. Optimal column configuration—experimental results.

Eight-column configuration; switching time = 65 s; $r(A)$ = 1.5 and $r(B)$ = 0.7; inlet mole fractions—propylene = 0.018 and dimethyl ether = 0.012.

A similar series of experiments was run with a three-column, single-desorbent configuration. Similar periodic concentration profiles were produced. The product stream impurities (propylene eluting at port B and dimethyl ether at port A) were slightly greater for the three-column configuration. The purity of propylene at port A was less than 85%.

The next design configuration, as shown in Figure 5, was to investigate the introduction of a separate carrier flow on desorption of the strongly sorbed component product stream (port B). Higher product purities were observed than those seen earlier, but additional improvements are possible.

The final three-desorbent design, shown in Figure 6, added an isolated purge gas stream before the feed column to remove all residual material. Experimental results from the four-column configuration are presented in Figures 8 and 9. Both desorbent carrier streams were matched at the same flow rate. The purge flow rate was increased by a factor of between 2 and 3.

Figure 8 shows the results for breakthrough ratios set at 1.5 and 0.7 for propylene and dimethyl ether, respectively. A total carrier flow of 800 mL/min was used. The feed flow rate for both solutes was 10 mL/min, giving feed mole fractions of 0.012. It is predicted that for an ideal dispersionless system the maximum observed concentrations at these breakthrough ratios will be three times the feed concentration for both components. Propylene behaves very near the ideal; three times the feed concentration was observed (0.036) over approximately one-third of the switching period. The strongly adsorbed component (dimethyl ether), however, did not behave ideally. Dispersion became a factor, and the material was distributed along the column. The propylene purity removed at port A exceeded 99.9%, and the dimethyl ether purity at port B also exceeded 99.9%.

Figure 9 shows the results for a switching period of 81 s and a carrier flow rate of 560 mL/min. The new single-column breakthrough time of propylene and dimethyl ether were approximately 60 and 128 s, respectively. Feed flow rates of 10 mL/min of both components were used, corresponding to mole fractions of 0.017. Many switches were used in these trials and the sampling period was offset from the switching period so that data were obtained every second into the switching period. The propylene purity exceeded 99.9%, while the dimethyl ether purity exceeded 91.5%.

A steady-state mass balance was calculated by summing the total steady-state mass of each component removed at each port during one period and dividing by the mass of each component added during that period. In excess of 99.9% of the propylene was recovered, while only 70% of the dimethyl ether was recovered. The impurity problems of the earlier configurations were eliminated and high throughput product streams were obtained, but the addition of the purge column is responsible for the loss of dimethyl ether. This can be attributed to the heterogeneity of the surface, which retains a portion of the dimethyl ether in the adsorbed state until the column in which it resides becomes the purge column. The extent of this loss depends upon the choice of the adsorbent and the tailing characteristics on that adsorbent.

The effect of the number of columns was evaluated further. The addition of a purge column raises to four the minimum number of columns necessary to perform the separation, but the existence of a maximum or optimal number of columns is unclear. To test this, a similar configuration was constructed with eight columns. Four additional columns were inserted between the feed column and the weakly sorbed component removal port.

In an ideal, dispersionless system, it is expected that the concentration profiles will be unaffected by the addition of extra columns. Separation occurs in the feed and the carrier columns, where the two components are present together. The product concentration profiles should not be altered by the presence of additional columns.

The experimental results show that the number of columns does affect the concentration profiles. Figure 10 displays results obtained with an eight-column configuration with $r(A) = 1.5$ and $r(B) = 0.7$. The inlet mole fractions were 0.018 and 0.012 for propylene and dimethyl ether, respectively. The total carrier flow was 800 mL/min. The propylene feedrate was 15 mL/min, and the dimethyl ether was fed at 10 mL/min. The predicted maximum concentration equals three times the propylene feed concentration over one-third of the switching cycle. Experimentally, the observed maximum concentration does not exceed two times the feed concentration. Varying operating conditions, such as altering flow rates and breakthrough ratios, produced similar results. In no circumstance does the maximum concentration exceed two times the feed concentration, unlike the four-column configuration, where three times the feed concentration was observed. To eliminate the possibility that this arose from column packing variations, the separator was reconfigured to eliminate the extra columns ahead of the feed column. The results from this four-column configuration reproduced the earlier four-column results, giving confidence that the column-packing technique was satisfactory, and that there was not sufficient variability in column packing to account for the observed deterioration of the concentrating effect in the eight-column separator. The optimal number of columns for this type of configuration then appears to be the minimum number—four columns—since the addition of extra columns accentuates dispersion effects.

Conclusions

The simulated countercurrent chromatographic separator is an efficient method for separating binary gas phase mixtures. Although the output does not mimic that of the true countercurrent chromatographic separator, two high purity product streams can be produced. The product concentration profiles are periodic at steady state. Concentrations exceed feed values during part of the switching cycle, and equal zero during the rest of the cycle. This may present advantages for dilute separations, if the control capabilities are such that the effluent product streams are stored during that part of the cycle where they exceed the feed concentration.

The choice of separator design depends upon the desired outcome. The final configuration arrived at in this investigation is best suited for obtaining high purity product streams. This design overcomes the problem caused by tailing, and gives significant improvement in product purity over the earlier design (Fish et al., 1993). However, the improved purity comes at the expense of a high carrier gas requirement and some loss of material. Less stringent purity requirements and an expensive carrier constraint might shift the optimal design to a single-desorbent configuration.

Optimization of the simulated countercurrent separator reveals several features. The optimal number of columns or sections within the system is also the minimum, four columns. The four-column optimal configuration contains three inlet carrier gas streams in addition to the binary feed gas and three outlet ports. The four columns are as follows: (1) an isolated purge column, immediately ahead of the feed column; (2) an isolated product-removal column, two columns behind the feed to remove the strongly sorbed product; (3) a main carrier gas column, one column behind the feed column; and (4) a feed column, where the weakly sorbed component elutes and is removed as a product stream. Each port is moved sequentially one column to the left at each switching time.

Several operational advantages exist for using the simulated countercurrent separator. High purity product streams (greater than 99.9%) are obtained while simultaneously increasing the throughput over traditional chromatographic separators. The "concentrating" effect allows for efficient separation of dilute binary mixtures. The final advantage is the ease at which this system can be operated relative to true countercurrent systems by eliminating the need for handling a moving-solids stream.

Acknowledgments

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