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Youichi Takahashi[a]; Shigeo Goto[a]
[a] DEPARTMENT OF CHEMICAL ENGINEERING, NAGOYA UNIVERSITY NAGOYA, JAPAN

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Continuous Separation of Fructooligosaccharides Using an Annular Chromatograph

YOUICHI TAKAHASHI and SHIGEO GOTO
DEPARTMENT OF CHEMICAL ENGINEERING
NAGOYA UNIVERSITY
NAGOYA, 461-01 JAPAN

ABSTRACT

A continuous rotating annular chromatograph with a rotating feed nozzle and product collectors was used to separate mixtures of fructooligosaccharides. Oligosaccharides could be continuously separated from monosaccharides and a disaccharide to obtain a lower calorie sweetener. Analytical solutions agreed well with experimental results.

INTRODUCTION

Continuous chromatographic systems have been developed for preparative separations. Moving chromatographic beds and simulated moving beds are mostly restricted to two-component separations (1). On the other hand, a continuous rotating chromatograph proposed by Martin (2) can be used to separate multicomponents. Begovich et al. (3) used a rotating annular bed with a stationary feed stream and a product collection port to separate metal ions.

In our previous papers (4–7), a continuous rotating annular chromatograph (CRAC) with a rotating feed nozzle and product collectors was proposed. This system is the opposite of the device of Begovich et al. (3). This system has the following advantages. Since a weighty bed need not be rotated, rotating power is saved and scaling up of the device is easy. The bed can be easily kept at constant temperature with a thermostat jacket. This is especially important for biological separations which re-

quire accurate temperature control. Three amino acids (aspartic acid, glutamic acid, and glycine) were separated by the difference of adsorption force for isocratic elution using CRAC (4). CRAC with two nozzles was used for nonisocratic elution to separate three amino acids (glutamic acid, glycine, and valine) (5). Also, a single component of an amino acid (valine) was concentrated continuously (6). Two components of proteins (myoglobin and hemoglobin) were separated and concentrated simultaneously using CRAC (7).

In this work, fructooligosaccharides mixtures are separated using CRAC. Fructooligosaccharides are produced from sucrose through enzymatic transfructosylation (8). Fructooligosaccharides, namely, 1-kestose (GF_2), nystose (GF_3), and 1-β-fructonystose (GF_4), are low calorie sweeteners and physiologically useful to improve the intestinal flora. On the other hand, monosaccharides, glucose (G) and fructose (F), and a disaccharide, sucrose (GF), are higher calorie sweeteners than are oligosaccharides. Therefore, these fructooligosaccharides need to be separated from monosaccharides (G and F) and disaccharide (GF).

EXPERIMENTAL

Operation

The CRAC apparatus used in this work was the same as described in a previous paper (4).

The cation exchange resin Dowex 50W-X4, in its sodium form (average diameter: 0.061 mm), was used as an adsorbent. A mixture of fructooligosaccharides (Meioligo-G, Meiji Seika Co. Ltd.) was used as a solute. The water content of the feed solution was 70 wt%. Table 1 indicates the composition of Meioligo-G. Distilled water was used as an eluent. The temperature of the annular bed was kept at 333 K. The concentrations of the components were measured by differential refractometer.

TABLE 1
Initial Conditions of Meioligo-G

	G	F	GF	GF_2	GF_3	GF_4
Weight %	14.6	15.4	11.6	26.4	27.1	4.9
C_0 (kg/m^3)	52.6	55.4	41.8	95.0	97.6	17.7

RESULTS AND DISCUSSION

Elution Curves in Conventional Column

Figure 1 shows the elution curves from a conventional column. The inside diameter of the column was 9 mm and the bed length was 450 mm. The void fraction, ϵ_B, in the bed was 0.381. GF_4 was first eluated, followed successively by GF_3, GF_2, GF, G, and F. The symbols in Fig. 1 indicate the experimental results. The solid lines indicate the results calculated by the analytical solution of Rasmuson (9). Kinetic parameters for the calculations were determined by the moment method (10). Adsorption isotherms were assumed to be linear. Elution curves for each component were measured by changing the eluent rates. The results of kinetic parameters are listed in Table 2. As the molecular weight increases, the adsorption equilibrium constant (K), intraparticle diffusivity ($\overline{D}$), and Peclet number (Pe) decrease.

Elution Curves in CRAC

Figure 2 shows the elution curves in CRAC. The abscissa in Fig. 2 shows the angular position from the nozzle (θ). The elution order was the same as in a conventional column. The peaks of G and F appear after two rotations from the feed nozzle. On the other hand, the peaks of GF_4, GF_3,

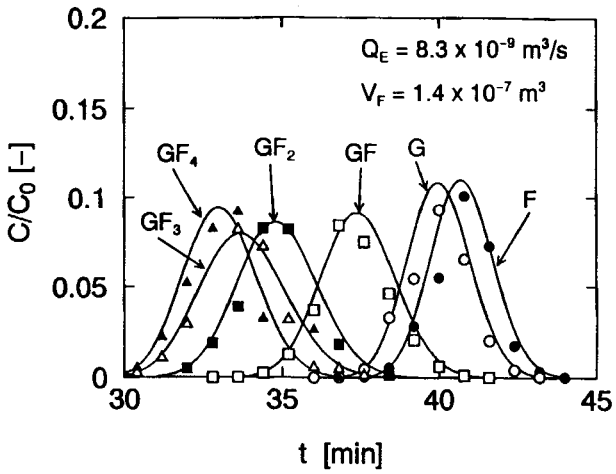

FIG. 1 Elution curves using conventional column.

TABLE 2
The Value of Parameters of Fructooligosaccharides Mixture

	Molecular weight	K (—)	$\bar{D} \times 10^{11}$ (m²/s)	Pe (—)
G	180.2	0.445	7.61	0.333
F	180.2	0.432	7.01	0.317
GF	342.3	0.364	4.85	0.143
GF_2	504.4	0.293	3.48	0.122
GF_3	666.6	0.261	2.50	0.110
GF_4	828.7	0.234	2.30	0.102

GF_2, and GF appear after one rotation. The monosaccharides (F and G) and fructooligosaccharides (GF_2, GF_3, and GF_4) separated well, but the disaccharide (GF) and other saccharides were not separated. The calculated results agreed very well with the experimental results.

The superficial velocity (u) and the injection time [t_0 ($= 2\pi Q_F/(Q_E + Q_F)\omega$)] in Fig. 2 were held at the same values as those ($t_0 = V_F/Q_E$) in Fig. 1. Therefore, the elution curves of CRAC can be predicted by experiments in a conventional column if time (t) is converted to θ/ω.

Figure 3 shows the elution curves found by using CRAC with another feed rate. The feed rate (indicated by Q_F) was increased by 4 times while

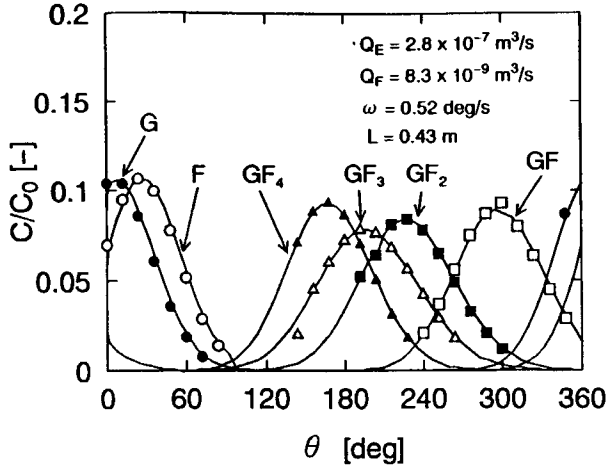

FIG. 2 Elution curves using CRAC at low feed rate.

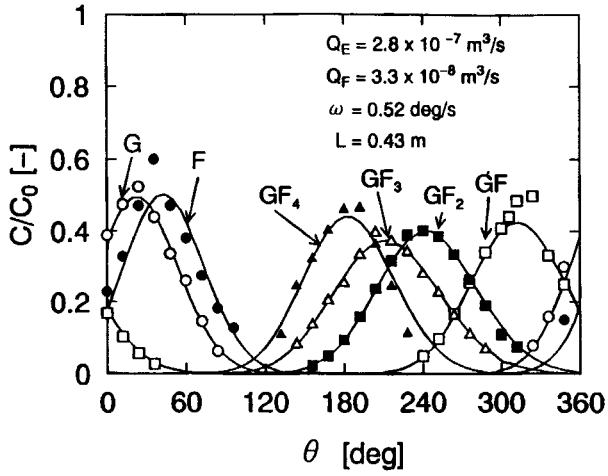

FIG. 3 Elution curves using CRAC at high feed rate.

the eluent rate (Q_E) and the rotating speed (ω) were held constant. Therefore, the peak concentration was increased as the feed rate (Q_F) increased. Therefore, the feed rate (Q_F) should be increased to recover higher concentrations.

Calculated Results of Longer CRAC Column

It is seen from Figs. 2 and 3 that the height of the experimental apparatus is insufficient to separate disaccharide (GF) from the oligosaccharides (GF_2, GF_3, GF_4) completely. Then simulation was done to improve separation.

Figures 4–6 shows the calculated results of a longer annular column with the same eluent and feed rate as in Fig. 3. As shown in Figs. 4–6, the bed length (L) was varied from 1.0 to 4.0 m, while the rotating speed (ω) was decreased to adjust the angular distance between the head and end of the elution curves to within 360 degrees. The purity of the oligosaccharides is 86 wt% at L = 1.0 m in Fig. 4 when the solution with angular coordinates from 100 to 250 degrees are collected. The purities at L = 2.0 and 4.0 m are 94 and 96 wt%, respectively. If the CRAC column is longer than 2 m, oligosaccharides (GF_2, GF_3, and GF_4) with purities greater than 94 wt% can be produced.

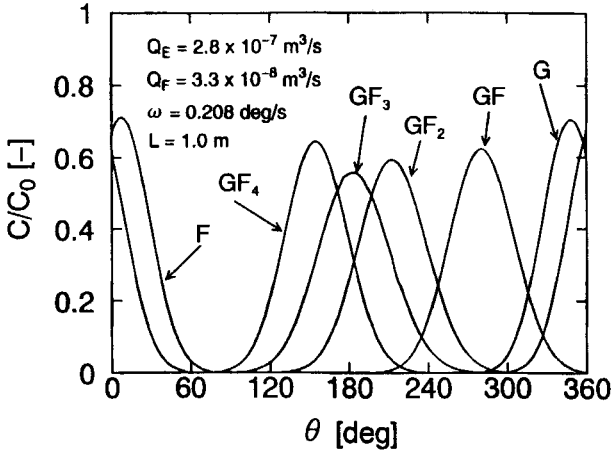

FIG. 4 Calculated results using CRAC at L = 1.0 m.

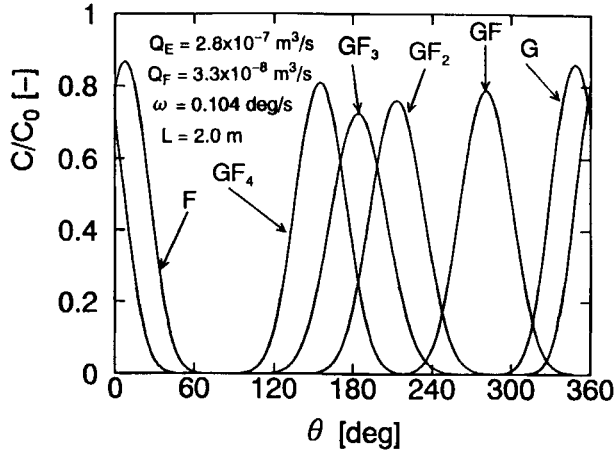

FIG. 5 Calculated results using CRAC at L = 2.0 m.

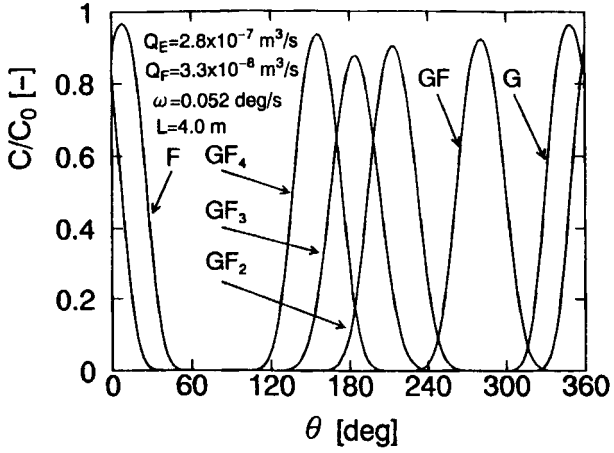

FIG. 6 Calculated results using CRAC at L = 4.0 m.

CONCLUSION

A mixture of fructooligosaccharides was separated by using CRAC. Monosaccharides and oligosaccharides could be separated completely. Disaccharides could not be separated from other components. Separation can be improved by using a longer annular column.

SYMBOLS

C	concentration in the liquid phase (kg/m^3)
C_0	feed concentration (kg/m^3)
$\overline{D}$	intraparticle diffusivity (m^2/s)
D_L	axial dispersion coefficient (m^2/s)
K	adsorption equilibrium constant (—)
L	bed length (m)
Pe	Peclet number ($= 2uR_P/\epsilon_B D_L$) (—)
Q_E	eluent rate (m^3/s)
Q_F	feed rate (m^3/s)
R_P	radius of adsorbent particle (m)
u	superficial velocity (m/s)
V_F	volume of pulse injection (m^3)

Greek

ϵ_B	interparticle void fraction in the bed (—)
θ	angular coordinate (deg)
ω	rotating speed (deg/s)

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