

# CONTINUOUS CHROMATOGRAPHIC SEPARATION OF PALATINOSE (6-*o*- $\alpha$ -D-GLUCOPYRANOSYL-D-FRUCTOSE) AND TREHALULOSE (1-*o*- $\alpha$ -D-GLUCOPYRANOSYL-D-FRUCTOSE) USING A SIMULATED MOVING-BED ADSORBER

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This paper deals with the continuous separation of palatinose (6-*o*- $\alpha$ -D-glucopyranosyl-D-fructose) and trehalulose (1-*o*- $\alpha$ -D-glucopyranosyl-D-fructose) using a simulated moving-bed adsorber. Palatinose is similar to sucrose in taste and food technological properties.<sup>3)</sup> Dentally it has low cariogenicity and may be anticariogenic.<sup>5)</sup> Trehalulose also has good sweetness and low cariogenicity.<sup>6)</sup> Palatinose is commercially produced from sucrose by action of  $\alpha$ -glucosyltransferase of fungi.<sup>4)</sup> As a by-product of the reaction a significant amount of trehalulose is formed.<sup>1)</sup> Both disaccharides are contained in palatinose molasses, which is residual mother liquor after separation of palatinose from the reaction mixture by crystallization. Separation of these

disaccharides may lead to better utilization of the molasses.

## 1. Experimental

The simulated moving-bed adsorber used had essentially the same configuration as an adsorber designed by Hashimoto *et al.*<sup>2)</sup> and manufactured by Shinko-Pfaudler Co., Ltd. A total of 16 columns were arranged circularly on a rotary valve consisting of two disks. The upper disk on which the columns were placed could be rotated 22.5° (360°/16) at regular intervals. The lower disk, to which the introduction ports of feed and desorbent and the withdrawal ports of extract and raffinate were attached, was stationary. The 16 columns on the disk were divided into four zones (zone I, II, III and IV) of four columns each at the four ports. Each column was  $1.38 \times 10^{-2}$  m in

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diameter and 0.10 m in length. Diaion UBK 530 resin of Ca-form (Mitsubishi Kasei Corporation; nominal particle size, 100 mesh) was used as adsorbent. Two types of solutions were used for the separation test. One was 4° Brix (solid content of 4%) model solution in which the concentrations of palatinose and trehalulose were each 2%. The other was 40° Brix palatinose molasses, prepared from commercial palatinose molasses by dilution. All the separation tests were carried out at 50°C.

## 2. Results and Discussion

A mathematical model of chromatographic separation using the simulated moving-bed adsorber has been developed by Hashimoto *et al.*<sup>2)</sup> The mass balance for component *k* in the adsorption columns is given by Eqs. (1) and (2).

$$\varepsilon \frac{\partial C_k}{\partial t} = -v_n \frac{\partial C_k}{\partial z} - Ka(C_k - C_k^*) \quad (1)$$

$$(1 - \varepsilon)m_k \frac{\partial C_k^*}{\partial t} = Ka(C_k - C_k^*) \quad (2)$$

Separation of palatinose and trehalulose can be simulated by numerical solution of these equations. Prior to the simulation, the constants contained in Eqs. (1) and (2) were determined. The distribution coefficients for palatinose and trehalulose estimated from the adsorption isotherms and the breakthrough curves were 0.242 and 0.307, respectively. The overall volumetric coefficient for both disaccharides was  $0.018 \text{ s}^{-1}$ , which was determined so that the experimentally obtained breakthrough curve and the elution curve might fit those obtained by simulation. The void fraction was estimated to be 0.366, the value evaluated for a bed packed with spheres of uniform particle size, because the adsorbent used was essentially spherical and very uniform in particle size (190–240  $\mu\text{m}$ ) on the basis of microscopic observation.

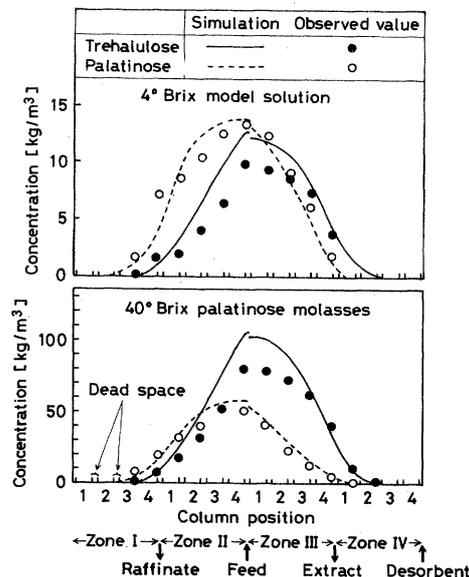
A separation profile of palatinose and trehalulose was obtained by a simulation model based on the separation parameters,  $\beta_{nk}$ , presented by Hashimoto *et al.*<sup>2)</sup> The separation profile did not agree with the experimental one. The parameter  $\beta_{nk}$  was then modified to  $\gamma_{nk}$ , taking account of the time lag of the liquid flow through the dead space between the columns.

$$\gamma_{nk} = \frac{v_n - (\varepsilon L + DS/S)/T}{(L/T)(1 - \varepsilon)m_k} \quad (3)$$

The chromatography was again simulated in consideration of Eq. (3). The criterion of  $\beta_{nk}$  for good separation<sup>2)</sup> was also applied to  $\gamma_{nk}$ . **Table 1** shows an example of the flow rates in each zone obtained on the basis of the criterion of  $\gamma_{nk}$  where a time interval of 130 s was chosen for rotation of the adsorption

**Table 1.** Flow rates for good separation

Flow rate (m/s)		Flow rate (m/s)	
$v_I$	$4.61 \times 10^{-4}$	$v_f$	$0.28 \times 10^{-4}$
$v_{II}$	$5.33 \times 10^{-4}$	$v_d$	$1.69 \times 10^{-4}$
$v_{III}$	$5.05 \times 10^{-4}$	$v_e$	$1.26 \times 10^{-4}$
$v_{IV}$	$6.31 \times 10^{-4}$	$v_r$	$0.71 \times 10^{-4}$



**Fig. 1.** Concentration profile of disaccharides in adsorption columns obtained by simulation based on  $\gamma_{nk}$  and experiment

columns. **Figure 1** shows the concentration profiles after a process time of 4 h at the flow rates shown in **Table 1**. The experimentally obtained data for 4° Brix model solution and 40° Brix palatinose molasses were in good agreement with the respective estimations by simulation. In the case of the palatinose molasses the ratio of palatinose/trehalulose, which was originally 1/2 in the feed solution, became 2/13 in the extract stream and 2/1 in the raffinate.

Consequently, it was found that continuous separation of palatinose and trehalulose could be well performed by using the simulated moving-bed adsorber.

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### Nomenclature

<i>a</i>	= specific particle surface area in bed	[m <sup>2</sup> /m <sup>3</sup> ]
<i>C</i>	= concentration in mobile phase	[kg/m <sup>3</sup> ]
<i>C*</i>	= concentration in equilibrium with <i>q</i>	[kg/m <sup>3</sup> ]
<i>DS</i>	= dead space between two adjacent columns	[m <sup>3</sup> ]
<i>K</i>	= overall mass transfer coefficient	[m/s]
<i>Ka</i>	= overall volumetric coefficient	[1/s]
<i>L</i>	= length of each adsorption column	[m]

$m$	= distribution coefficient	[—]
$S$	= cross-sectional area of adsorption columns	[m <sup>2</sup> ]
$T$	= interval for rotation of adsorption columns in simulated moving-bed adsorber	[s]
$t$	= time	[s]
$v$	= superficial liquid flow velocity	[m/s]
$z$	= axial distance	[m]
$\beta_{nk}$	= parameter presented by Hashimoto <i>et al.</i> <sup>2)</sup>	[—]
$\gamma_{nk}$	= parameter defined by Eq. (18)	[—]
$\epsilon$	= void fraction of bed	[—]
<b>&lt;Subscript&gt;</b>		
$d$	= desorbent	
$e$	= extract	
$f$	= feed	
$k$	= arbitrary component	
$n$	= zone number (I to IV)	

$r$  = raffinate

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