CONTINUOUS SEPARATION OF GLYCEROL AND NaCl WITH LINEAR AND UNFAVORABLE ADSORPTION ISOTHERMS BY USE OF A SIMULATED MOVING-BED ADSORBER

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Key Words: Simulated Moving Bed, Unfavorable Adsorption Isotherm, Continuous Separation, Ion Exclusion Chromatography

Introduction

Simulated moving-bed adsorbers have been successfully used to separate continuously two components with linear or favorable adsorption isotherms.[1-6] The adsorber can be utilized, in principle, for a binary separation system with any type of isotherms. In this work we demonstrated the continuous separation of glycerol and NaCl by using an adsorber packed with a strongly acidic cation-exchange resin (Amberlite HFS-471X with 8% DVB content) of sodium ion form. The adsorption isotherm of NaCl on the resin is unfaborable.

1. Adsorption Isotherms and Mass Transfer Coefficients

The adsorption isotherms at 303 K of NaCl and glycerol on the resin were obtained by analyzing their breakthrough curves measured at various influent concentrations. In the binary system, the amount adsorbed of an adsorbate was calculated from its breakthrough curve, which was measured after the bed was equilibrated with a 1000 mol/m^3 solution of another adsorbate. The bed voidage was determined from the response curve of a pulse input of soluble starch with a molecular weight of more than 10^4.

Received April 20, 1992. Correspondence concerning this article should be addressed to K. Hashimoto.

Figure 1 shows the adsorption isotherms. The isotherm of glycerol was linear, while that of NaCl was unfavorable. The isotherms were independent of each other because the results for each adsorbate in single and binary systems were represented by a curve for both the adsorbates. The amount adsorbed q was empirically related to the equilibrium concentration C by $q_G = 3.23 \times 10^{-4} C_G$ and $q_N = 1.06 \times 10^{-6} C_N^{1.72}$

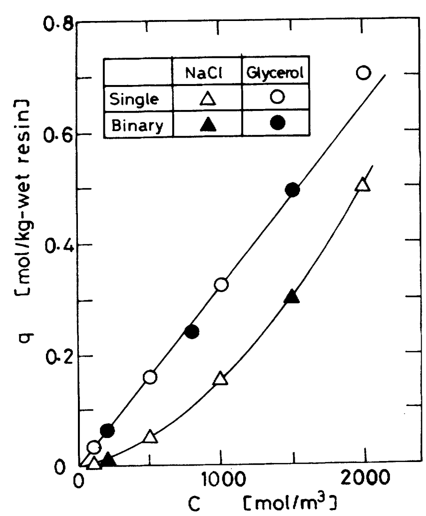

Fig. 1. Adsorption isotherms of NaCl and glycerol on Amberlite HFS-471X with 8% DVB content of sodium ion form at 303 K

for glycerol and NaCl respectively. When NaCl solution was introduced to the bed, bed shrinkage occurred. In the calculations of q and the overall mass transfer coefficient, the height of the shrunken bed was used. The unfavorable adsorption isotherm of NaCl is due to ion exclusion.

The overall volumetric mass transfer coefficients were determined so that the calculated breakthrough curves may fit to the experimental ones. The broadening of the breakthrough curves of glycerol could be expressed by using a parameter $K_f a_v$ under the assumption of a plug flow. On the other hand, it failed to express the broadening of the curves of NaCl by a parameter. Therefore, axial dispersion was taken into consideration for NaCl. In the calculation of the curves for NaCl, the Peclet number was assumed to be 0.5. As shown in **Fig. 2**, the dependencies of the coefficient on the concentration and the flow rate were not significant in the ranges examined. The estimated value of $\overline{K_f a_v}$ of NaCl was $8.3 \times 10^{-3}\,\mathrm{s}^{-1}$, and that of $K_f a_v$ of glycerol was also $8.3 \times 10^{-3}\,\mathrm{s}^{-1}$. The solid and broken curves in the figure are the calculated ones using these values.

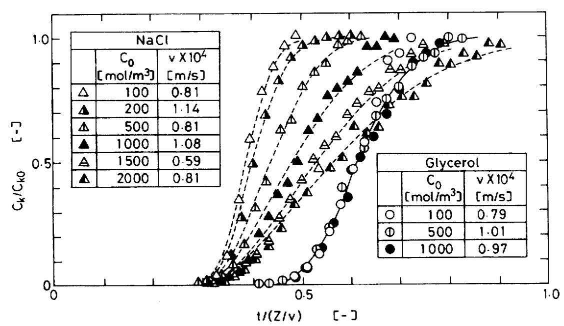

Fig. 2. Breakthrough curves for NaCl and glycerol in a bed packed with Amberlite HFS-471X of sodium ion form

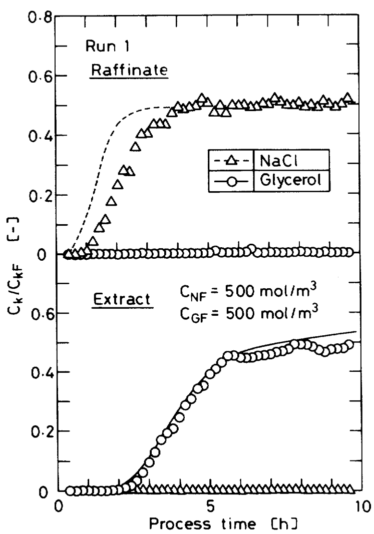

Fig. 3. Transient changes in concentration of NaCl and glycerol in raffinate and extract streams in run 1

2. Continuous Separation of Glycerol and NaCl

A simulated moving-bed adsorber with four zones was used for the continuous separation of the adsorbates. The adsorber comprised 12 columns, and the apparatus was the same as that used previously.[5] The liquid emerging from zone I (the effluent stream defined in the previous paper[5]) was not recycled to the desorbent stream, so as to confirm that it contained neither of the adsorbates. **Figures 3** and **4** show the transient changes in the concentrations of NaCl and glycerol in the raffinate and extract streams in runs 1 and 2, respectively. The experimental runs were conducted at different feed concentrations. Details of the operating conditions are listed in **Table 1**. In both runs the adsorbates were almost completely separated, and the purities of NaCl in the raffinate stream and

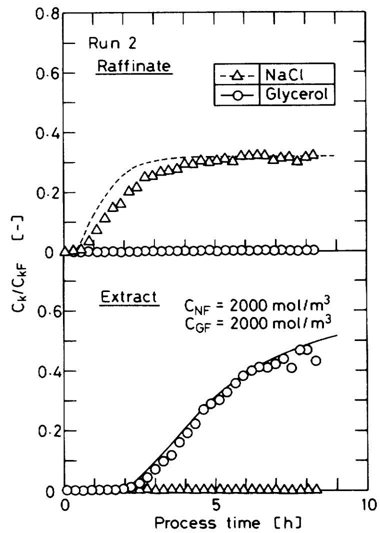

Fig. 4. Transient changes in concentration of NaCl and glycerol in raffinate and extract streams in run 2

Table 1. Operating conditions for the continuous separation of NaCl and glycerol by simulated moving-bed adsorber*

	Run No.	1	2
Inner diameter of a column	[m]	1.5×10^{-2}	1.5×10^{-2}
Bed height in each column** (average)	[m]		
Initial		0.179	0.179
Final		—	0.175
Feed concentration of			
NaCl	[mol/m^3]	500	2000
glycerol	[mol/m^3]	500	2000
Interval of valve rotation	[s]	720	477
Flow rate	[m^3/s]		
Feed		0.47×10^{-8}	0.33×10^{-8}
Desorbent		3.65×10^{-8}	5.18×10^{-8}
Raffinate		0.80×10^{-8}	0.53×10^{-8}
Extract		0.80×10^{-8}	0.51×10^{-8}

* The adsorber had four zones, each of which included three columns.
** The bed voidage was 0.368 for both runs.

glycerol in the extract stream were both over 99% on a molar basis. It was also confirmed that the concentrations of the adsorbates in the effluent stream were negligibly low, although the results are not shown. The solid and broken curves in the figures are those calculated by the intermittent moving-bed model,[5] in which some modifications were made. In run 1 the initial bed height was used in the calculation because of negligible bed shrinkage. On the other hand, the bed shrinkage could not be ignored in run 2. The height averaged over the shrunken beds was used in the calculation. The calculated curves coincide well with the experimental results.

Conclusion

The simulated moving-bed adsorber could be successfully utilized to separate NaCl and glycerol continuously, even though one of them exhibits an unfavorable adsorption isotherm. It was also shown that the intermittent moving-bed model proposed previously[4,5] can be used to predict transient changes in the concentrations of adsorbates in the raffinate and extract streams.

Nomenclature

C	= concentration	[mol/m^3]
$K_f a_v$	= overall volumetric mass transfer coefficient of glycerol	[s^{-1}]
$\overline{K_f a_v}$	= overall volumetric mass transfer coefficient of NaCl	[s^{-1}]
q	= amount adsorbed	[mol/kg]
t	= time	[s], [h]
v	= superficial velocity	[m/s]
Z	= bed height	[m]

〈Subscripts〉

F	= feed
G	= glycerol
k	= arbitary component (glycerol or NaCl)
N	= NaCl
0	= influent

Literature Cited

1) Barker, P. E., F. J. Ellison and B. W. Hatt: *Ind. Eng. Chem. Process Des. Dev.*, **17**, 302 (1978).
2) Broughton, D. B., R. W. Neuzil, J. M. Pharis and C. S. Bearly: *Chem. Eng. Prog.*, **66**(9), 70 (1970).
3) Ching, C. B.: *J. Chem. Eng. Japan*, **16**, 49 (1983).
4) Hashimoto, K., S. Adachi, H. Noujima and H. Maruyama: *J. Chem. Eng. Japan*, **16**, 400 (1983).
5) Hashimoto, K., M. Yamada, Y. Shirai and S. Adachi, *J. Chem. Eng. Japan*, **20**, 405 (1987).
6) Maki, H., H. Fukuda and H. Morikawa: *J. Ferment. Technol.*, **65**, 61 (1987).