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Experimental Studies on the Degree of Separation in Thermal Diffusion Columns

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Abstract

Data were obtained in a flat-plate continuous-flow thermogravitational column to check the theories developed by Furry et al., and Yeh and Chu. Separations of benzene–*n*-heptane mixtures were measured, with flow rate, feed concentration, and temperature difference being varied in the experiments. Yeh and Chu's work are in excellent agreement with the experimental data, while the agreement of Furry et al.'s work with the experimental results is fair for equifraction solution and poor for high and low values of feed concentration.

INTRODUCTION

The principles of thermal diffusion in thermogravitational column were discerned by Clusius and Dickel (*3, 4*). The first complete presentation of the theory was that of Furry et al. (*9, 10*). In their report, transport equation was developed by considering the net transport of one component of a binary system passing through a plane perpendicular to the walls of a thermal diffusion column. Integration of the transport equation and incorporation of the appropriate boundary conditions together with several approximations yield

$$H\left[c(1-c) - \frac{\sigma}{H}(c_T - c)\right] = K\frac{dc}{dz} \tag{1}$$

for the enriching section and

$$H\left[c(1-c)+\frac{\sigma}{H}(c_B-c)\right]=K\frac{dc}{dz} \tag{2}$$

for the stripping section. The transport constants in Eqs. (1) and (2) are defined by

$$H=\alpha\beta_T\rho g(2\omega)^3B(\Delta T)^2/(6/\mu\overline{T}) \tag{3}$$

$$K=\beta_T^2\rho g(2\omega)^7B(\Delta T)^2/(9/D\mu^2)+2\omega DB\rho \tag{4}$$

Equations (1) and (2), associated with the boundary conditions

$$Z=L_e, \qquad c=c_T \tag{5}$$

$$Z=-L_s, \qquad c=c_B \tag{6}$$

were solved by Furry et al. under the following assumptions:

$$c(1-c)\cong 0.25, \qquad \text{i.e., } 0.3<c<0.7 \tag{7}$$

$$L_e=L_s=L/2 \tag{8}$$

The solution is

$$\Delta_2=c_T-c_B=\frac{H}{2\sigma}[1-\exp(-\sigma L/2K)] \tag{9}$$

Numerous investigators (*1, 2, 5–8, 11–21, 23, 24*) have made attempts to improve upon the theory. However, the product of the concentration dependence of the thermal diffusion effect incorporated into Eqs. (1) and (2) was assumed to be constant in all previous works, and the results obtained under this assumption are still not valid for $c<0.3$ or $c>0.7$. Yeh and Chu (*22*) derived a generalized equation of separation applicable to the whole range of concentration in thermal diffusion columns by linear approximation. It is the purpose of this work to check the theories developed by Furry et al. and Yeh and Chu by the experimental results.

METHOD OF LINEAR APPROXIMATION

Yeh and Chu (*22*) pointed out that the product form of the concentration dependence of the thermal diffusion effect incorporated in Eqs. (1) and (2) can be approximated by a straight line over the whole range of concentration

$$c(1-c)=a+bc \tag{10}$$

with the appropriate choice of constants:

$$a = c_i^2 - \frac{\Delta^2}{8} \tag{11}$$

$$b = 1 - 2c_i \tag{12}$$

Substituting Eq. (10) into Eqs. (1) and (2), integrating, and making use of the condition $c = c_i$ at $z = 0$, gives

$$\Delta_1 = c_T - c_B = -S + \sqrt{S^2 + 8c_i(1 - c_i)} \tag{13}$$

in which

$$S = \frac{2\{\sigma' L' + 2(1 - 2c_i)L' e^{-[2(1-2c_i)L' + \sigma' L']}\} \{\sigma' L' - 2(1 - 2c_i)L' e^{[2(1-2c_i)L' - \sigma' L']}\}}{L'\{2\sigma' L' + [2(1 - 2c_i)L' - \sigma' L']e^{-[2(1-2c_i)L' + \sigma' L']} - [2(1 - 2c_i)L' + \sigma' L']e^{[2(1-2c_i)L' - \sigma' L']}\}} \tag{14}$$

$$\sigma' = \frac{2\sigma}{H} \tag{15}$$

$$L' = \frac{HL}{4K} \tag{16}$$

Equation (13) is the correct form of Eq. (25) in Yeh and Chu's work which contains a printing error.

All that has been shown in Yeh and Chu's work is that Eq. (13) appears better than Eq. (9) for high and low values of c_i. To establish the validity of Eq. (13) to the whole range of c, comparisons with experimental data should be made.

EVALUATION OF CONSTANTS *H* AND *K*

For a given system, H and K are constants which may be found from Eqs. (3) and (4). Unfortunately, some of the physical constants required for the evaluation of H and K are not really available. These two transport coefficients can be most easily evaluated from the experimental data as follows.

$0.3 \leq c \leq 0.7$

If Eq. (9) is applied to two sets of data with the same value of feed concentration, with the condition that $\sigma_B = 2\sigma_A$, then

$$\Delta_A = \frac{H_I}{2\sigma_A}\left[1 - \exp\left(-\frac{\sigma_A L}{2K_I}\right)\right] \tag{17}$$

$$\Delta_B = \frac{H_I}{2\sigma_B}\left[1 - \exp\left(-\frac{\sigma_B L}{2K_I}\right)\right] \tag{18}$$

Solving Eqs. (17) and (18) for H and K, since $\sigma_B = 2\sigma_A$, gives

$$H_I = \frac{\sigma_A \Delta_A^2}{\Delta_A - \Delta_B} \tag{19}$$

$$K_I = \frac{\sigma_A L}{2 \ln\left(\frac{\Delta_A}{2\Delta_B - \Delta_A}\right)} \tag{20}$$

$0 \leq c \leq 1$

It is readily shown that Eq. (14) reduces, if σ' is sufficiently large, to $S = \sigma'$. Then Eq. (13) becomes

$$\Delta_1' = -\sigma' + \sqrt{\sigma'^2 + 8c_i(1 - c_i)} \tag{21}$$

or

$$H = \frac{4\sigma\Delta_1'}{8c_i(1 - c_i) - \Delta_1'^2} \approx \frac{\sigma\Delta_1'}{2c_i(1 - c_i)} \tag{22}$$

If (H_I, K_I) and (H, K) represent two sets of transport coefficients with different feed concentrations, then from Eqs. (3) and (4)

$$\frac{H}{H_I} = \left[\frac{\mu T_m}{\beta_T \rho (\Delta T)^2}\right]_I \left[\frac{\beta_T \rho (\Delta T)^2}{\mu T_m}\right] \tag{23}$$

$$K = K_I \left[\frac{\rho D (\Delta T)^2}{H^2 T_m^2}\right]_I \left[\frac{H^2 T_m^2}{\rho D (\Delta T)^2}\right] \tag{24}$$

In obtaining Eqs. (23) and (24) we assumed that $\alpha_I \approx \alpha$, $\bar{T} \approx T_m$, and the ordinary diffusion term in transport direction ($2wDB\rho$) in Eq. (4) is negligible.

EXPERIMENT

A vertical flat-plate, thermogravitational thermal diffusion column was constructed with two smooth 20 by 100 by 0.6 cm stainless steel plates

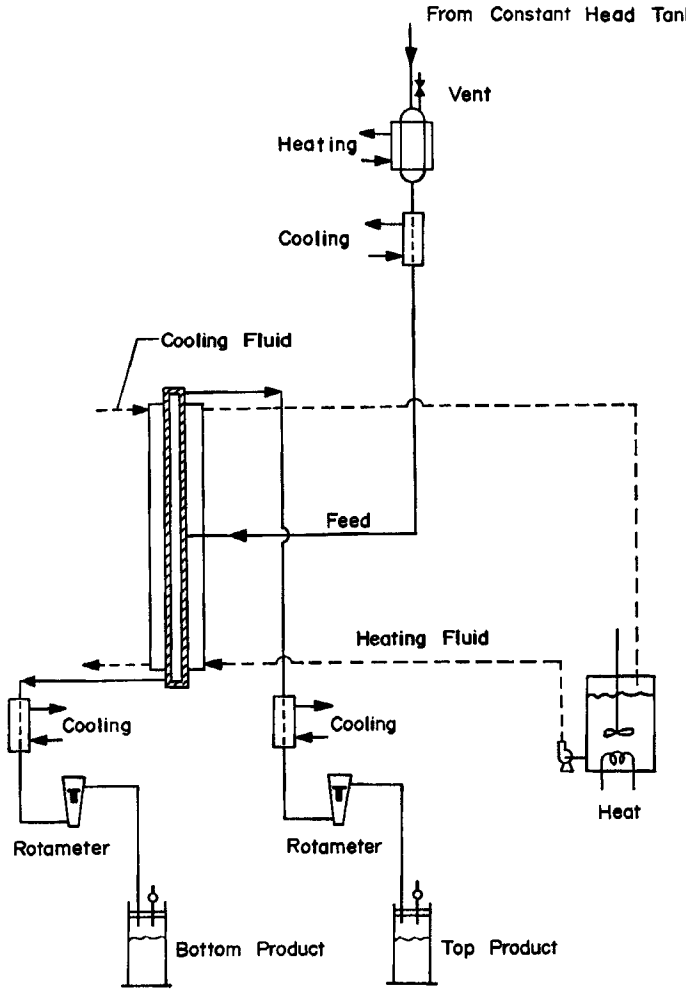

FIG. 1. Flow diagram of flat-plate continuous-type thermal diffusion column.

for the hot and cold surfaces. A 0.033 cm aluminum metal sheet with a 9.6 by 89.6 cm area cut out for the column space was sandwiched between the hot and cold plates. Hot and cold water were circulated through the jackets on each side, countercurrent to each other. The rates of circulation were sufficiently high to assume that the temperature change from the inlet to the outlet was not more than 10°F in either stream. Four copper constantan thermocouples located on the surface of each plate were used to measure the surface temperature. These thermocouples were installed as follows: A small hole drilled through the side of the hot or cold plate was joined by another hole drilled perpendicular to the surface of the plate. A small thermocouple wire was put through with its tip flush with the surface, and fixed in position with soft solder. Excess solder was then carefully sanded off. The mean temperatures of the cold and hot plate were 99.5 and 157.5°F, respectively, for $c_i = 0.1$ and 0.5; and 98 and 146°F, respectively, for $c_i = 0.7$ weight fraction benzene. The plate temperatures were kept constant within 0.3°F at all times. No variation in temperature along the width of the plate was observed.

The column was connected as shown in Fig. 1. The feeds, consisting of benzene and *n*-heptane with various compositions, were introduced from a constant-head tank into the feed line joined to a small channel similar to used that for the thermocouples in the cold plate and located half way between the bottom to the top of the column. Top and bottom products were withdrawn continuously at the same constant rate and passed through cooling coils and rotameters to the product accumulators. Samples of both streams were analyzed at 30 min intervals with a Bausch and Lomb Abbe refractometer with a sodium lamp until the steady-state was reached as indicated by no change in the refractive index over a period of 2 hr. Extra pure, reagent grade benzene and *n*-heptane were used to prepare the feed. The refractive index was measured with a precision corresponding to $\pm 0.1\%$ in composition.

RESULTS

The experimental data obtained are shown in Table 1. Since duplicate runs made with $c_i = 0.5$ were in excellent agreement with each other, the data at this value of feed concentration were considered to be the most reliable, and the system constants H_I and K_I were evaluated from the two data point of this set as obtained in the low rate region with a flow rate of two. The experimental quantites are:

TABLE 1

Comparison of Separation Obtained from Experimental Work and Theoretical Equations

σ (g/min)	Δ_0 (%)	Δ_1 (%)	Δ_2 (%)	Conditions and quantities	
0.079	13.5	10.5	36.6	$c_l = 0.1$	
0.162	10.5	8.6	29.9	$T_m = 588.5°R$	$\Delta T = 58°F$
0.320	7.2	6.7	21.5	$\rho = 0.817$ g/cm^3,[a]	$D = 2.65 \times 10^5$ cm^2/sec[a]
0.641	4.8	4.3	12.7		
1.305	2.5	2.3	6.4	$H = 0.1676$ g/min	$K = 11.187$ (g)(cm)/min
2.011	1.5	1.5	4.2		
0.067	39.5	37.0	39.7	$c_l = 0.5$	
0.135	29.5	28.3	29.5	$T_m = 588.5°R$	$\Delta T = 58°F$
0.270	19.0	18.0	18.3		
0.551	9.6	9.5	9.5	$\rho = 0.733$ g/cm^3,[a]	$D = 3.05 \times 10^5$ cm^2/sec[a]
1.167	4.7	4.5	4.5	$H_I = 0.105$ g/min	$K_I = 4.252$ (g)(cm)/min
2.443	2.2	2.1	2.1		
0.064	25.0	30.0	39.1	$c_l = 0.7$	
0.129	19.5	19.9	24.0	$T_m = 582°R$	$\Delta T = 48°F$
0.261	10.0	10.6	12.7	$\rho = 0.70$ g/cm^3,[a]	$D = 3.6 \times 10^5$ cm^2/sec[a]
1.093	2.7	2.6	3.0	$H = 0.0667$ g/min	$K = 2.195$ (g)(cm)/min
1.750	1.6	1.6	1.9		

[a]These physical properties are taken from the *Journal of Chemical and Engineering Data, 16*(4), 425 (1971).

$$\sigma_A = 0.067 \text{ g/min}, \qquad \Delta_A = 0.395 \text{ wt fraction } n\text{-heptane}$$

$$\sigma_B = 0.135 \text{ g/min}, \qquad \Delta_B = 0.295 \text{ wt fraction } n\text{-heptane}$$

$$L = 89.6 \text{ cm}, \qquad c_i = 0.5, \qquad \Delta T = 58°\text{F}$$

Substitution of these values into Eqs. (19) and (20) gives

$$H_I = 0.105 \text{ g/min} \tag{25}$$

$$K_I = 4.252 \text{ (g)(cm)/min} \tag{26}$$

The values of H for $c_i = 0.1$ and 0.7 were evaluated from Eq. (22) with the experimental data obtained at the highest flow rates, respectively, as shown in Table 1. Then the values of K were calculated from Eq. (24) by using the appropriate physical quantities listed in Table 1. The results are:

$$H = 0.1676 \text{ g/min}, \qquad K = 11.187 \text{ (g)(cm)/min for } c_i = 0.1$$

and

$$H = 0.0667 \text{ g/min}, \qquad K = 2.195 \text{ (g)(cm)/min for } c_i = 0.7$$

The value of H_I for $c_i = 0.5$ was also checked by Eq. (22), and the result (0.107 g/min) was found very close to that shown in Eq. (25). Furthermore, all the values of H and K for each c_i were substituted into Eq. (14) to make sure that $S \approx \sigma'$ at the highest values of σ.

From the above values the separations were calculated from the corresponding separation equations. The results are also presented in Table 1.

CONCLUSION

The comparisons of the results obtained by Yeh and Chu and Furry et al. with those from the experimental work are shown in Table 1. It was found that the experimental results for the system benzene–*n*-heptane were in excellent agreement with those obtained by Yeh and Chu from linear approximation, while the agreement of experimental results with those obtained by Furry et al. was good for $c_i = 0.5$, poor for $c_i = 0.7$, and very poor for $c_i = 0.1$. It was also found that the agreement of experimental results with those obtained by Furry et al. appears better than with those obtained by Yeh and Chu for $c_i = 0.5$. This is reasonable since Eq. (9) is valid for equifraction solution and the system constants H_I and K_I were determined from Eq. (9). However, the degree of separations obtained by Furry et al. were greater than those obtained by Yeh and Chu and those from the experimental results, and the differences between

these separations increase as the values of $|c_i - 0.5|$ increase. This is also reasonable since $c(1 - c)$ decreases as c varies from 0.5, while Furry et al. assumed that $c(1 - c) = 0.25$, the maximum value of $c(1 - c)$. Equation (13) appears better than Eq. (9) for high and low values of c_i. As shown in the previous work (*22*), thermal diffusion for $c_i = 0.1$ and 0.3 correspond to those for $c_i = 0.9$ and 0.7, respectively. Consequently, the validity of Eq. (13) for the whole range of concentration has been established.

SYMBOLS

a	a constant, defined by Eq. (11)
B	column width
b	a constant, defined by Eq. (12)
c	fraction of component 1 in a binary solution
c_B, c_T	fraction of component 1 in the product stream exiting from the stripping, enriching section
c_i	fraction of component 1 in the feed stream
D	ordinary diffusion coefficient
g	gravitational acceleration
H	system constant, evaluated by Eq. (3)
H_I	H for equifraction solution
K	system constant, evaluated by Eq. (4)
K_I	K for equifraction solution
L	total column length
L_e, L_s	column length in the enriching, stripping section
L'	dimensionless column length, $HL/4K$
S	system constant, defined by Eq. (14)
T	absolute temperature
T_m	$(T_1 + T_2)/2$
T_1, T_2	temperature of cold, hot wall
$\overline{T}$	reference temperature
ΔT	$T_2 - T_1$
z	transport direction

Greek Letters

α	thermal diffusion constant
β_T	$-(\partial\rho/\partial T)$
Δ	$c_T - c_B$

Δ_0	Δ obtained from experimental work
$\Delta_1, \Delta_2, \Delta_1'$	Δ obtained from Eqs. (13), (9), (21)
μ	viscosity
ρ	mass density
ω	one-half of the distance between the plates of a thermal diffusion column
σ	average mass flow rate, $(\sigma_e + \sigma_s)/2$
σ_e, σ_s	mass flow rate from the enriching, stripping section
σ'	$2\sigma/H$

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