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Some Liquid Thermal Diffusion Separations in a Continuous Thermogravitational Column

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Some Liquid Thermal Diffusion Separations in a Continuous Thermogravitational Column

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Abstract

The separation of three binary mixtures on *n*-heptane–1,1,2,2-tetrachloroethylene, *n*-propanol–1,1,2,2-tetrachloroethylene, and *n*-heptane–methylcyclohexane was studied in a continuously operated thermogravitational column. The effects of feed rate, the ratio of top and bottom product rates, and temperature difference on separation were determined. The separations were found to be dependent on the feed rate with the maximum separation occurring at flow rates approaching zero. Over the range studied, the effect of top to bottom product flow rate ratio was found to be negligible. Temperature difference appeared to influence separation when finite flow rates were used.

INTRODUCTION

Much of the published work on liquid separation by thermal diffusion is concerned with batch operation of thermogravitational columns. Due to the very small capacity of the usual concentric cylinder columns, batch operation is not a commercial prospect except in certain rare situations. If the commercialization of the process is to be achieved, even for high priced materials, attention must be paid to continuous separation. Little work in this area has been reported in recent years.

Jones (*1*) extensively investigated the separation of liquids in continuous flow thermogravitational columns and discussed its dependence on column length, annular spacing, and temperature difference. Trevoy and Drickamer (*2*) worked with binary mixtures of paraffin hydrocarbons and benzene. Powers and Wilke (*3*) used a flat plate apparatus for continuous flow studies to confirm the theory developed by Furry, Jones, and Onsager (*4*) and

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proposed modifications to the theory. They studied the influence of flow rate, column length, temperature difference, and surface spacing and inclination of the surfaces on the separation obtained. Heines, Larson, and Martin (5), using concentric cylinder columns, studied the effects of some of these variables and feed composition on the separation produced.

BASIC THEORY

By taking into account the relevant mass and heat transfer equations, Furry, Jones, and Onsager (*4*) developed a theory for the operation of a thermal diffusion column. For batch operation the following transport equation was obtained:

$$\tau = HC(1 - C) - K\frac{dc}{dz} \tag{1}$$

where τ is the total transport of the component concentrating at the top of the column and C is its mass fraction; z is the coordinate parallel to the surfaces of the column (x may be defined as the coordinate normal to the surfaces). H, the transport coefficient due to thermal diffusion, is defined as

$$H = \frac{\alpha\beta\rho g B(\Delta T)^2(2a)^3}{6!T_{\mathrm{av}}\mu} \tag{2}$$

and

$$K = K_c + K_d \tag{3}$$

where K_c is the transport coefficient due to convection defined as

$$\frac{\beta^2\rho g^2 B(\Delta T)^2(2a)^7}{9!D\mu^2} \tag{4}$$

and K_d as

$$(2a)\rho DB \tag{5}$$

The equilibrium separation factor q may be defined by

$$q = \frac{C_{T\infty}/(1 - C_{T\infty})}{C_{B\infty}/(1 - C_{B\infty})} \tag{6}$$

Integrating over the column length at steady-state conditions yields

$$\ln q = \frac{H}{K} L \tag{7}$$

where L is the total length of column.

The theory may be extended to continuous operation in which the feed is introduced at the center of the column and the products are removed from the top and bottom (*6*).

For this situation, Eq. (1) becomes

$$\tau = W_T C + HC(1 - C) - K \frac{dc}{dz} \tag{8}$$

where W_T is the top product flow rate.

A material balance over the top part of the column gives

$$\tau = W_T C_{T\infty} \tag{9}$$

A combination of Eqs. (8) and (9) gives

$$W_T(C_{T\infty} - C) = HC(1 - C) - K \frac{dc}{dz} \tag{10}$$

Similarly for the bottom section of the column,

$$W_B(C - C_{B\infty}) = HC(1 - C) - K \frac{dc}{dz} \tag{11}$$

where W_B is the bottom product flow rate.

Integration of Eqs. (10) and (11) gives a complex result difficult to utilize. However, if $C(1 - C)$ is assumed to be a constant, a simpler result is obtained, i.e.,

$$e^{L W_T W_B / K(W_T + W_B)} = \frac{HC(1 - C)}{HC(1 - C) - \dfrac{W_T W_B}{F}(C_{T\infty} - C_{B\infty})} \tag{12}$$

where F is the feed rate.

If a center feed is employed:

$$L_T = L_B = \frac{L}{2} \tag{13}$$

and for a special case where the product rates are equal:

$$W_T = W_B = \frac{F}{2} \tag{14}$$

For $C(1 - C)$ constant and batch operation:

$$\Delta^1_\infty = \frac{H}{K} LC(1 - C) = C^1_{T\infty} - C^1_{B\infty} \tag{15}$$

Simplification of Eq. (12) still further gives

$$\frac{\Delta_\infty}{\Delta^1_\infty} = \frac{1 - e^{-(FL/4K)}}{FL/4K} \tag{16}$$

Equation (16) provides a comparison of the separation obtained in a continuous column to the separation in the column operated batchwise as a function of the feed rate, the column length, and the variables found in the transport coefficient K defined in Eq. (4).

EQUIPMENT AND PROCEDURE

A concentric cylinder thermogravitational column was used to investigate continuous separation of three binary mixtures. The column was made from two accurately machined stainless steel tubes concentrically arranged. The inner tube was cooled by the circulation of thermostatically controlled water through it. The outer tube was heated electrically by resistance wire helically wound round the tube. The column length (L) was 1.27 m, the spacing between the columns ($2a$) was 0.309 mm, and the "column width" (B) was 89.743 mm.

Two thermocouples were positioned at the inlet and outlet of the cold water and other thermocouples were located in the top, center, and bottom of the outer tube wall so that an estimate of the hot surface temperature could be made. The feed port was located midway along the vertical column length and the outlet ports were located at the top and bottom of the column.

Figure 1 shows the general layout of the equipment used in the experiments. The apparatus is fully described by Bourkiza (7).

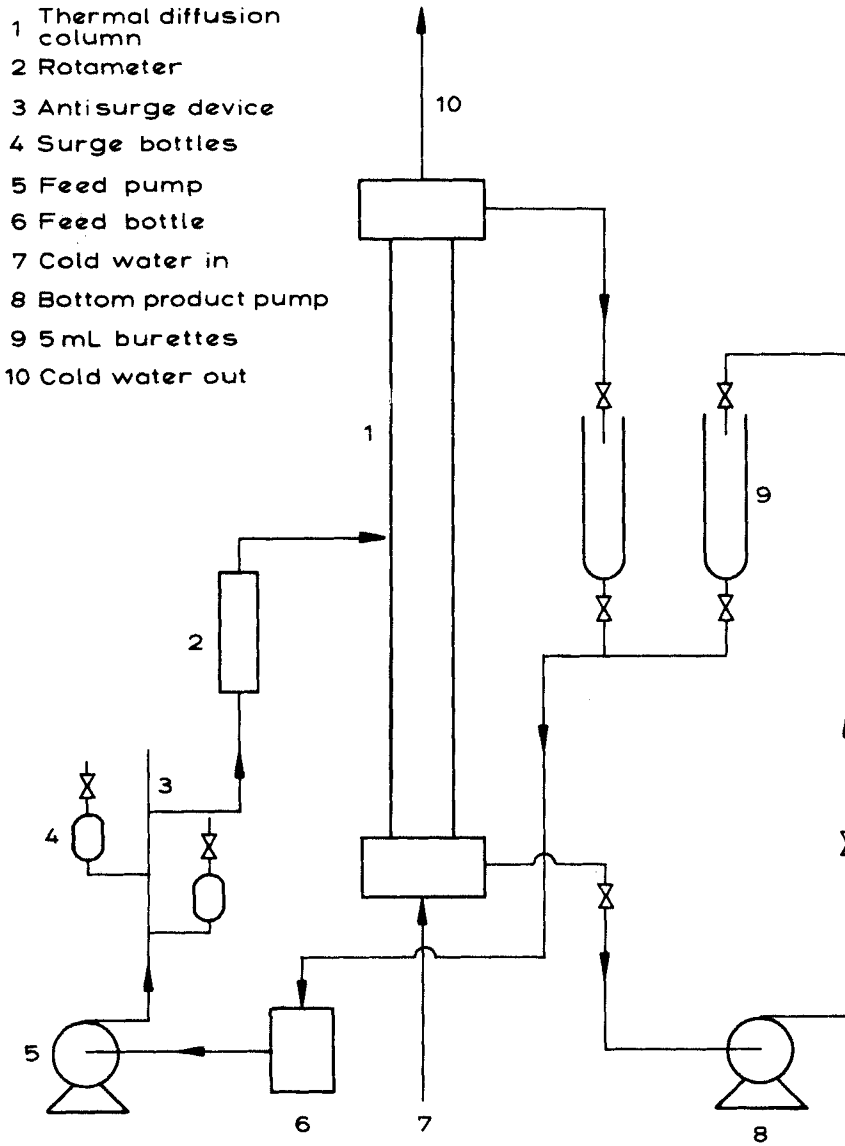

FIG. 1. Center feed thermogravitational column flow diagram.

At the beginning of a run, the cold water stream was turned on and the heating current in the outer tube windings was switched on, so that the desired temperature difference was obtained (in these experiments, the set ΔT was within the range 19 to 26 K). The annulus and all the lines in the equipment were purged with the test mixture to be used in the particular experiment. A rotameter was used to establish an approximate flow rate through the column. The two 5-mL burettes were used to measure the flow rates at the top and bottom by timing the volumetric flow. Control was achieved by adjustment of the valves in the lines.

The column was allowed to reach steady-state conditions before any data were recorded. The compositions of the feed and the top and bottom products were measured by refractive index.

The behavior of three binary mixtures was examined in the test apparatus. The mixtures were *n*-heptane–1,1,2,2-tetrachloroethylene, *n*-propane–1,1,2,2-tetrachloroethylene, and *n*-heptane–methylcyclohexane, giving combinations of straight chains and a ring structure.

RESULTS AND DISCUSSION

The effect of temperature difference (ΔT), feed flow rate (F), and the top and bottom product rates ratio W_T/W_B were investigated.

Changes in separation due to variation of the ratio of the top and bottom product ratio W_T/W_B were studied for all three binary mixtures. The results are plotted on Figs. 2–4, in which separation is plotted against W_T/W_B. Different feed rates were used for each mixture in order to get some qualitative information for the effect of feed rate. The data obtained suggest that for W_T/W_B within the range 0.3 to 5 and the feed rate constant, the separation is independent of the top to bottom product ratio. If the product off-take rates are increased to high values but the ratio W_T/W_B is still within the range, the separation will tend to zero, i.e., the residence time within the column is the overriding parameter.

The results for the three systems used in these studies agree well with the data of Heines et al. (5) for the system benzene–*n*-heptane.

Although not completely conclusive, the trends in Figs. 2–4 suggest that variations in W_T/W_B can be neglected.

Figure 5 shows the effect of feed rate on separation for the system *n*-heptane–methylcyclohexane. The drastic effect of increased flow rate confirms the theory represented by Eq. (16). With the small annular space (0.309 mm) in the column, low flow rates would be required in order to achieve a reasonable degree of separation. The data were obtained with W_T/W_B approximately 1 although there was a variation over the range 0.99

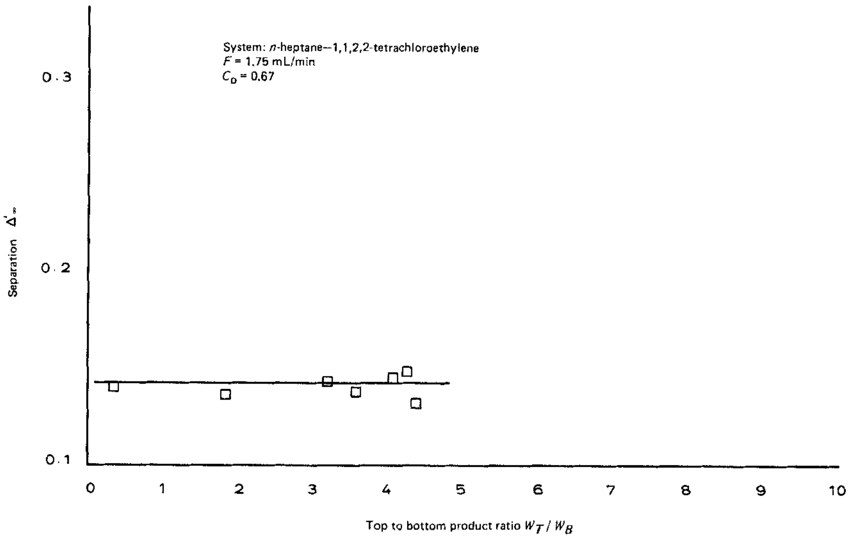

FIG. 2. Effect of ratio of top to bottom product flow on separation.

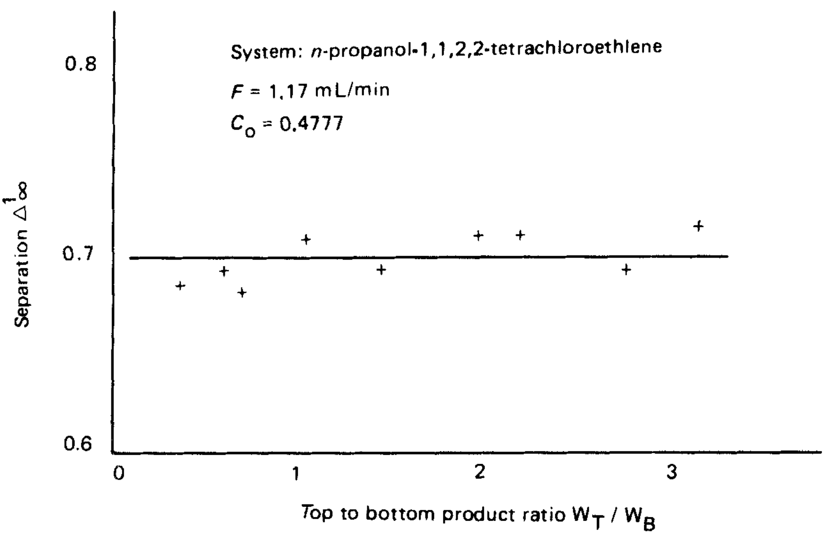

FIG. 3. Effect of ratio of top to bottom product flow on separation.

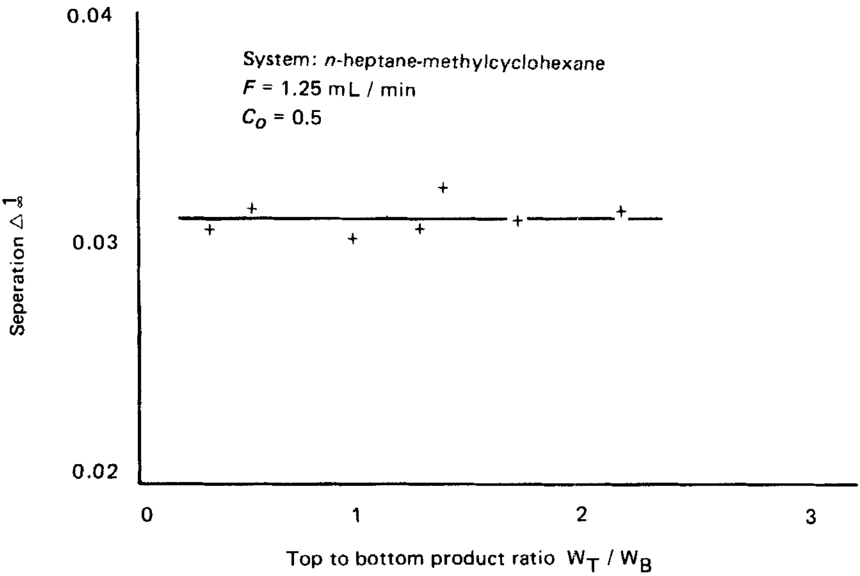

FIG. 4. Effect of ratio of top to bottom product flow on separation.

to 1.15 due to difficulties in the control of the small flow rates used in the experiments.

Studies on the effects of temperature difference on separation were made using the system *n*-heptane–methylcyclohexane under steady-state conditions. According to the theory for columns operated continuously, the temperature difference should be accounted for in the group $FL/4K$ since K, the transport coefficient, includes a term dependent upon ΔT. However, a definite grouping according to ΔT can be discerned despite some scatter of results.

The theory for batch operation (or total reflux) predicts that separation is independent of ΔT for steady-state conditions. Extrapolation of the data back to zero flow rate on Fig. 5 would lead to the conclusion that the separation was independent of ΔT since at this condition the differences would represent a small fraction of the overall separation. As flow rate is increased, the effect of differences in ΔT becomes more pronounced.

CONCLUSIONS

For the conditions for continuous operation studied, separation is independent of the top to bottom product ratio. The maximum separation

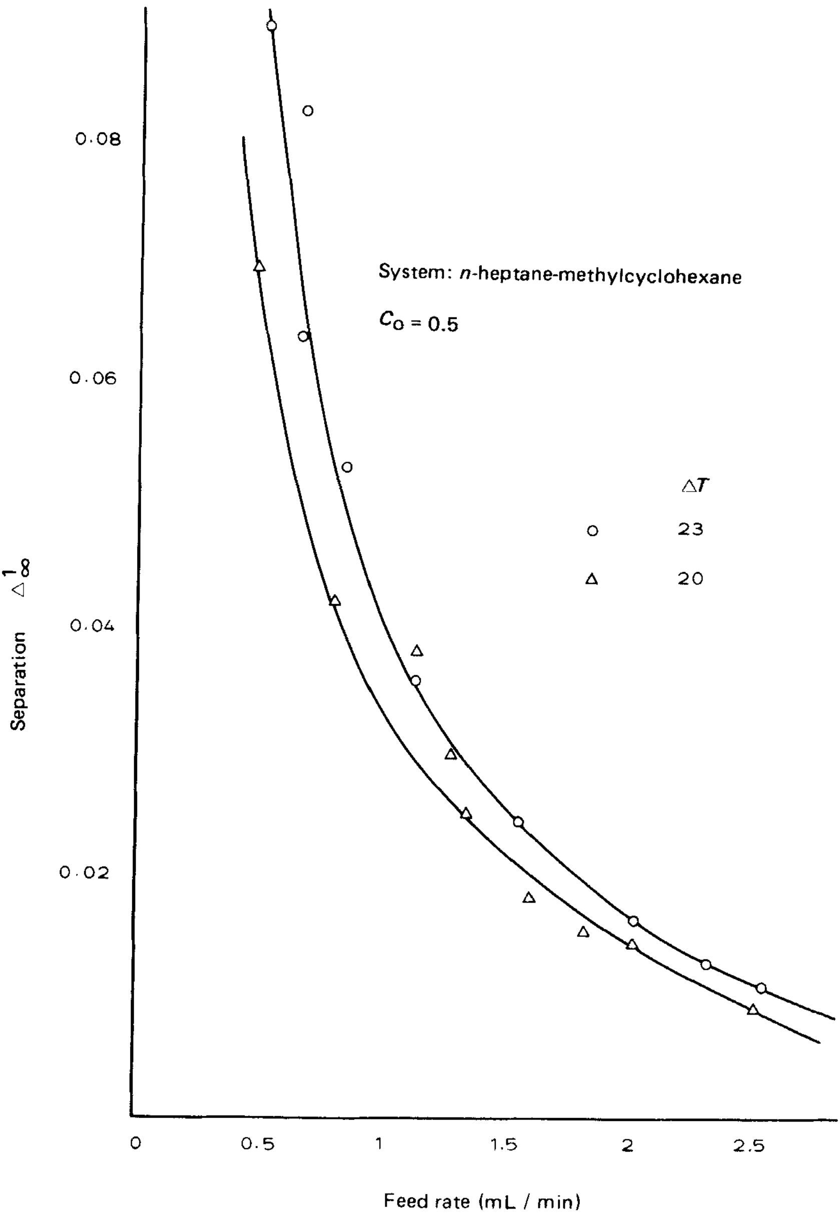

FIG. 5. Effect of temperature difference on separation at a feed concentration of 0.5.

occurs at zero flow rate. Temperature difference has an increasing effect on separation as the flow rate is increased (in contrast to batch operation where separation is independent of temperature difference).

SYMBOLS

a	one-half of the annulus width
B	column width in the Y-direction
C	mass fraction concentration of the reference component
D	ordinary diffusion coefficient
F	feed rate to the column
g	gravitational acceleration
H	transport coefficient defined by Eq. (2)
K_c	transport coefficient defined by Eq. (4)
K_d	transport coefficient defined by Eq. (5)
L	column length
T	absolute temperature
T_{av}	average absolute temperature
ΔT	temperature difference between the hot and cold walls of the column
W	product flow rate
X, Y, Z	coordinate directions

Greek Symbols

α	thermal diffusion factor
β	temperature coefficient of density $(\partial\rho/\partial T)$
ρ	density of the mixture
Δ_∞	$C_{T\infty} - C_{B\infty}$ batch separation
Δ^1_∞	$C^1_{T\infty} - C_{B\infty}$ continuous separation
μ	viscosity of the mixture
ρ	density
τ	total transport of the reference component

Subscripts

T	top
B	bottom
∞	steady-state

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