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A Method of Improving Liquid Thermal Diffusion Separations

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NOTE

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Abstract

By the use of a permeable, but immiscible, liquid layer in a liquid thermal diffusion column, it is possible to reduce remixing effects and hence improve separation. The technique is illustrated by using water as the permeable layer in the separation of benzene and carbon tetrachloride.

INTRODUCTION

In a thermogravitational thermal diffusion column, the conventional column, the continuous cascading associated with the vertical natural convection, together with the horizontal concentration gradient produced by the thermal diffusion effect, promote the establishment of a vertical concentration profile within the annular space (*1*). For a binary mixture this means that one component tends to concentrate at the top whereas the other component enriches the mixture at the bottom of the column.

The amount of separation attainable is, however, limited by the hydrodynamic process itself since the vertical convection also has a remixing effect which counteracts the separation promoted by the cascading (*2*). The remixing becomes more important as the separation proceeds and, ultimately, a dynamic equilibrium is established between demixing and remixing and no further separation takes place: the separation is, then, at the steady-state for which the following diagrammatic equation applies:

$$\mathrm{TB} \underset{\text{remixing}}{\overset{\text{demixing}}{\rightleftarrows}} \mathrm{T} + \mathrm{B}$$

The situation is better understood with the help of Fig. 1 where the principal fluxes within the annulus are represented. Assuming that component T concentrates at the top and B at the bottom, the net effects on the regions in which the column is divided are:

Region HT: Concentration (of component T at the top).
Region TC: Remixing (component T is brought from a region of highly concentrated T to a region of less concentrated T).
Region CB: Concentration (of component B at the bottom).
Region BH: Remixing (component B goes from a region of high concentration to a region less concentrated in B).

It is thus clear that any improvement in the equilibrium separation must be associated with either an increase of the cascading effects or a reduction of the remixing, which implies a "protection" of the concentrated zones (top and bottom). In terms of the above equation this means that the equilibrium represented must be moved toward the right-hand side.

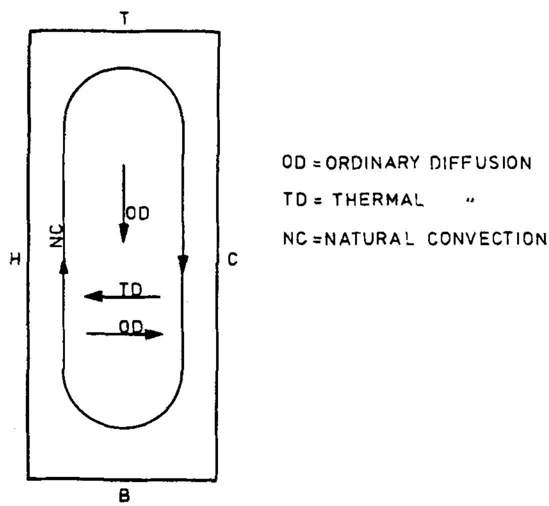

FIG. 1. Model of thermal diffusion column.

The relative increase of the cascading has been the object of most of the modified column designs that have been proposed in the literature as in the case of packed (*3*), tilted (*4*), wired (*5*), and rotary (*6*) columns.

The method or process suggested in the present paper has a different approach: the aim is not so much the increase of the cascading but rather the relative reduction of the remixing effects.

THE METHOD

One of the physical properties that may be used to follow the separation of a binary mixture in a thermal diffusion column is the density. In most cases the more dense component concentrates at the bottom and only then can the present process be used.

As the separation proceeds, a vertical gradient of density builds up, i.e., the densities at the top and bottom of the column, d_t and d_b "move away" from the feed mixture density, d_0, and at the equilibrium they lie somewhere between d_0 and the pure-component densities as shown in Fig. 2.

Suppose, now, that before feeding the mixture into the column an immiscible liquid layer of density d_l such that $d_0 < d_l < (d_b)_{eq}$ is placed at the bottom of the column (Fig. 3*a*). The separation proceeds normally until $d_b = d_l$, but a further increase in separation produces a situation in which $d_b > d_l$ and therefore a certain amount of mixture (rich in B) passes through the bottom layer (as outlined in Fig. 3*b*). The process continues and the column is virtually divided into two distinct sections, the upper section rich in T and the lower section rich in B. Both of the sections undergo the cascading–remixing effects and most probably exchange material between them through the permeable layer (see Fig. 3*c*). The steady state is reached when the vertical concentration profile over the entire length of the column is unchanged.

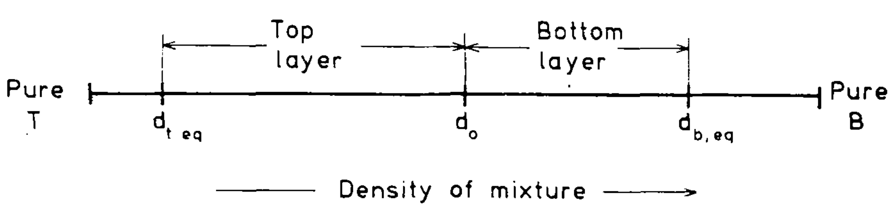

Fig. 2. The density distribution.

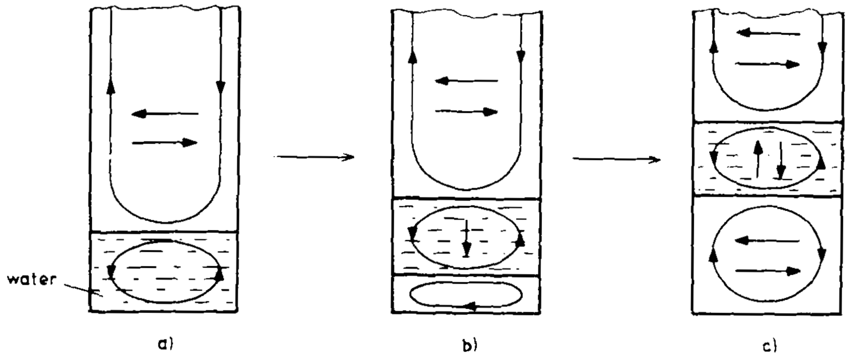

FIG. 3. Development of the process.

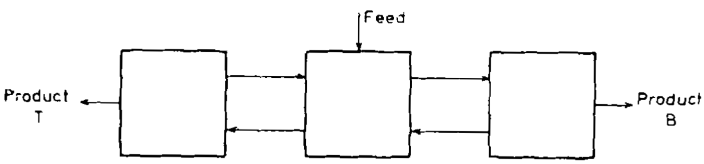

FIG. 4. Three-column cascade analogy.

The final height of the two sections is primarily a function of the location of d_l relative to d_0 and $(d_b)_{eq}$.

Naturally the permeable layer can be located, initially at the top, in which case the layer density must be $d_0 > d_l > (d_t)_{eq}$, the process remaining essentially the same.

It is interesting also to note that the process described has some analogies with a three-column cascade of the type shown in Fig. 4.

EXPERIMENTAL

The process was tested with mixtures of benzene–carbon tetrachloride and a water liquid layer whose height was less than 10% of the full height of the column. For the "top layer" the feed composition was, in molar fraction C_6H_6, $c_0 = 0.684$, which corresponds to a density smaller than the density of the water layer. For the "bottom layer" the feed composition was $c_0 = 0.844$, the density then being slightly higher than that of the water layer.

The apparatus consisted of a concentric cylinder column with an annulus width of 0.057 cm and a total height of 102 cm along which there were 11 sampling ports (extremes included) equally spaced and 3 feed ports at the top, bottom, and at the center.

The heating and cooling of the column walls was made by water flow from thermostatic baths. Each wall had a series of four independent copper–constantan thermocouples placed along the length to measure the wall temperature and to provide information regarding the homogeneity of the wall temperature. In the present work the temperature difference between the walls was 18 ± 0.5°C and the mean temperature was 27 ± 2°C.

The technique is simple: the mixture was fed through the bottom of the column to ensure the displacement of air from the annular space, the more dense of the immiscible liquids (mixture or layer) being fed in last.

The rate of sampling was zero, i.e., only one sample was taken per run, in accordance with the recommendations of Vichare and Powers (*7*). After withdrawing the sample the column was discharged, washed, and a fresh mixture fed into the apparatus for a new run.

The analysis of the mixture composition was made by refractometry, the accuracy being 0.0002 units of the refractive index, which corresponds to 0.001 units of the molar fraction of the mixture.

Further details of the equipment and techniques are to be found elsewhere (*8*).

The results obtained, shown in Table 1 and Fig. 5, clearly indicate that, in fact, the use of an immiscible liquid layer increases the degree of separation obtainable, the other variables being kept constant.

The shape of the curves in Fig. 5 agrees well with the theoretical "picture" described before: the presence of the water layer only affects the separation after the vertical concentration profile has reached the point where $d_b = d_l$.

TABLE 1

Experimental Data

Feed mixture composition (molar fraction of benzene)	Water layer		Steady-state separation (%)
	Initial location	Height (% column height)	
0.684	—	—	55
0.684	Top	8	66
0.844	—	—	44
0.844	Bottom	5	48

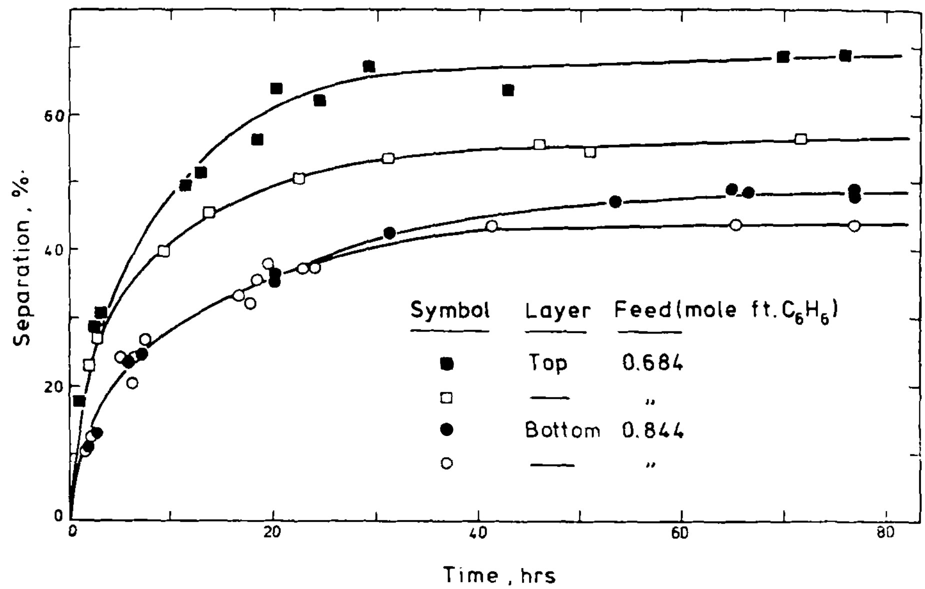

FIG. 5. Experimental time–separation curves for two different feed compositions with and without the use of a water layer.

CONCLUDING REMARKS

(a) The proposed process of operation improves separation without requiring any alteration in the basic design of the column or increasing the separation time.

(b) Further studies are required to define the best characteristics of the liquid layer. Points to be considered must include the location of d_l relative to d_0 and $(d_b)_{eq}$ [or $(d_l)_{eq}$], the effect of the layer height, the influence of the surface tension of the liquids, the simultaneous use of a top and a bottom layer, and the possibility of using the process in continuous operation.

(c) In principle, the method described may be used in the apparatus whose design is cascade-improving.

(d) A possible difficulty of the process appears to be the problem of selecting the substance for the liquid layer which must not contaminate the separation products.

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