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GAS SEPARATION BY A CONTINUOUS MEMBRANE COLUMN

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ABSTRACT

The continuous membrane column provides a revolutionary new separation technique. In gaseous diffusion the continuous membrane column is used to separate as highly concentrated products both the most permeable and least permeable gases from a feed mixture of any composition. The main features of the column are countercurrent enrichment, high reflux and minimal backmixing. The new method eliminates the need for numerous interstage compressors and extensive product stream recycling found in conventional gaseous diffusion cascades.

Experiments are carried out for systems of CO_2-O_2, O_2-N_2 (air), and CO_2-N_2 mixtures using continuous membrane columns made out of silicone rubber membrane. Also, a theoretical model is developed to interpret the experimental data. The agreement between theory and experiment is excellent. The maximum degree of separation can be achieved at total reflux. It is experimentally verified that the maximum degree of enrichment attainable by a conventional method can easily be exceeded without limit when a continuous membrane column is employed.

Finally, a comparative study has been conducted for a conventional gas permeator and a continuous membrane column.

INTRODUCTION

Gas separation by means of membrane permeation has been recognized for almost 150 years. However, its industrial

applications have been rather spotty. Well known examples are uranium isotope separation at Oak Ridge, hydrogen purification with palladium alloy membrane by Union Carbide, and separations of industrial gases by Du Pont's Permasep. Like all the other membrane processes, gas separation by membrane depends on the permselectivity of the membrane. If the perfect membrane can be found then one-step separation is possible. However, only partial enrichment can be achieved by use of a conventional permeator. When further separation becomes necessary, multiple stages must be cascaded in series. This requires a cumbersome operation and prohibitively high cost.

The recent development of hollow fiber membranes improved significantly the permeator design, construction, and operation. Various model calculations have confirmed the experimental results (1-5). Axial pressure loss (2,6-9), capillary deformation (4,6,10), pressure dependency of gas permeability (11-14), flow patterns (15), two-membrane cells (3,16-18), and separations with cascades (2,16-23) have been studied. Comprehensive reviews dealing with capillary permeators can be found in books by Hwang and Kammermeyer (24) and Meares (25). Still, the permeation cell is widely regarded as a single-stage device. There is an absolute limit (determined by the permselectivity) in attainable enrichment by a single permeator.

The objective of this paper is to introduce the newly developed "continuous membrane column" to gas separations and compare its performance with that of a conventional gas permeator. No longer is the gaseous permeation cell operated as a single stage, but rather as a continuous cascade (26,27). When a binary feed mixture is introduced to the continuous membrane column, nearly complete separation can be achieved on a continuous basis. The inherent feature of countercurrent plug flow operation creates a strong internal reflux action which polarizes the most and least permeable components along the membrane. In spite of the differences between the separation mechanisms for membrane

and equilibrium processes, the performance characteristics of a continuous membrane column are very similar to those of packed distillation, extraction, and gas absorption columns.

As in the packed columns of equilibrium processes, there are two streams in a continuous membrane column. One travels upward and the other moves downward. However, unlike in the packed columns, these two streams are separated by a permselective membrane. A feed stream is introduced centrally into the column and products are withdrawn at the ends as shown in Fig. 1. As the mixture moves downward along the high-pressure side of the membrane, the more permeable gas preferentially permeates through the membrane leaving the concentrated less permeable gas behind. At the bottom of the column, the high-pressure stream is stripped

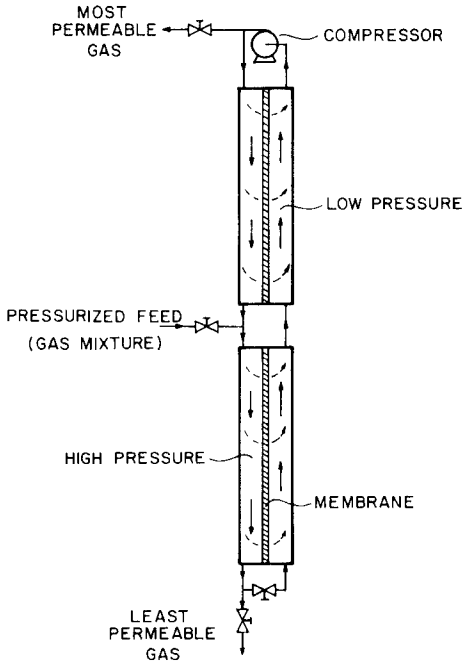

FIGURE 1 Continuous Membrane Column for Gas Separation

of the more permeable component yielding a concentrated product of the less permeable gas. On the other hand, the low-pressure stream travels upward being enriched with the more permeable component. At the top of the column, the concentration of the more permeable gas reaches a maximum. A compressor recycles the low-pressure stream to the high-pressure side at this point, thus completing the necessary reflux action. A top product can be withdrawn at the compressor end. The column can be divided into an enriching section and a stripping section. These sections may be operated individually if so desired.

Thus, development of the continuous membrane column represents a dramatic change in membrane separations. The conventional cascade with multistage cells and numerous interstage compressors are not required in order to increase the degree of separation.

THE CONTINUOUS MEMBRANE COLUMN

The first paper (26) dealing with a continuous membrane column reported the experimental results for several gaseous binary systems. These included the stripper, enricher, and total column data, with and without product removal. A satisfactory numerical simulation was also developed to interpret the experimental data. It was proposed to characterize continuous membrane columns by the number of membrane units (NMU) and the height of a membrane unit (HMU), defined analogously to NTU and HTU.

In the present paper, some additional data for several binary systems are reported in order to demonstrate how the continuous membrane column works. The experimental apparatus and procedures are the same as previously reported. Each permeation cell consists of a bundle of 35 silicone rubber capillaries (0.0238 cm I.D. x 0.0610 cm O.D.) made by Medical Engineering Corp., Racine, Wisconsin.

1. Total Reflux

When no products are removed from either the stripper or the enricher, a total reflux condition similar to that in the equilibrium processes is achieved. The degree of concentration variation along the column reaches a maximum if the column is operated at total reflux. Note that under total reflux conditions there is a feed input into the individual stripper or enricher, however, no feed into a total membrane column.

When a stripper is operated at total reflux with zero bottom product flow rate, the bottom composition must necessarily become zero. This is illustrated in Fig. 2 for the CO_2-O_2 system. The solid curves represent calculated profiles using the same computer program as the one described previously (26). The theoretical profiles agree very well with point samples taken along the shell-side of the column. The feed flow rates, which are the same as reject flow rates, for the 89.2% and 62.4% CO_2 runs were 0.956 and

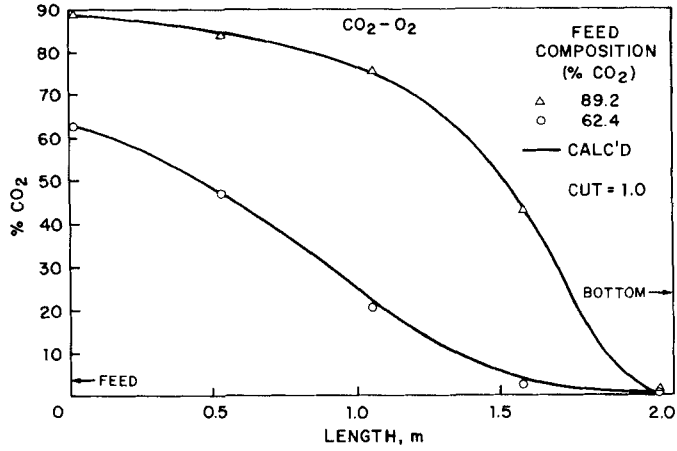

FIGURE 2 Shell-Side Composition Profiles for CO_2-O_2 in a Total Reflux Stripper

0.517 std cc/sec, respectively. The feed pressures were 168.5 and 166.2 cm Hg, respectively.

Results for the operation of the enricher for the O_2-N_2 system are shown in Fig. 3. In order to study the effect of feed flow rate on the concentration variation, the feed flow rate was changed in increments of about 0.05 std cc/sec. The output pressure of the compressor was comparable in each case ranging from 159 to 168 cm Hg. The results show that the degree of oxygen enrichment increases as the net amount of flow through the column decreases. This is because the amount of permeation becomes greater relative to the feed flow rate. During the start-up period for a column operated at total reflux, the less permeable gas builds up in the stripper and the more permeable gas accumulates in the enricher. Oxygen in the pressurized mixture preferentially permeates through the membrane and is swept back to the compressor by the countercurrent low pressure stream. This action lets oxygen accumulate within the column until a steady-

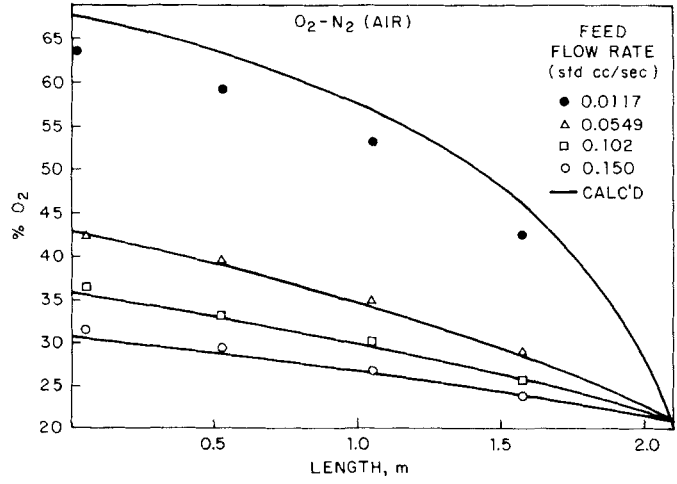

FIGURE 3 Shell-Side Composition Profiles in a Total Reflux Oxygen Enricher

state condition is reached. By reducing the feed flow rate, the relative amount of oxygen-enriched permeate entering the compressor increases resulting in even greater oxygen accumulation.

2. Total Membrane Column with Products

The individual stripper and enricher units can be combined to form a total continuous membrane column as shown in Fig. 1. Whereas only a small fraction of a binary feed mixture can be separated as a concentrated product with an individual stripper or enricher, the entire feed stream can be separated into two constituent components by a total membrane column. The unconcentrated product from the stripping section serves as an internal feed to the enriching section and vice versa. An external feed is introduced between the sections, and concentrated products are removed at opposite ends of the column on a continuous basis. Thus, operation of the total membrane column parallels that of continuous equilibrium processes such as distillation and extraction.

A feed mixture of 54.8% CO_2 and 45.2% N_2 was introduced into a 5.12 m membrane column at 0.237 std cc/sec. The top product contained 94.5% CO_2 at 0.108 std cc/sec and the bottom product consisted of 82.2% N_2 at 0.129 std cc/sec. The compressor load was 1.96 std cc/sec. Axial pressure loss inside the capillaries was less than 4% of the absolute inlet pressure. The calculated shell-side composition profile agreed very well with the experimental data as shown in Fig. 4. This system illustrates that very substantial separation can be achieved continuously by a small-scale membrane column with moderate compressor load. However, it should be noted that the separation factor is large for this system (11.6).

As the separation factor decreases, the degree of concentration variation decreases as demonstrated by the O_2-N_2 (air) system in Fig. 5. The separation factor for O_2-N_2 is 2.018. Room air was fed into a 4.24 m membrane column to produce 36.8%

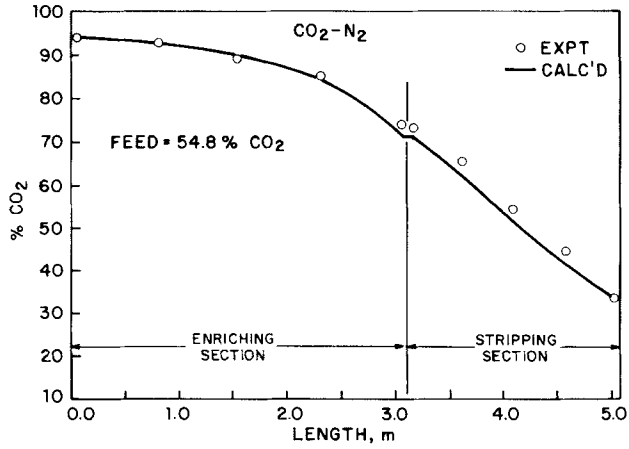

FIGURE 4 Shell-Side Composition Profile for CO_2-N_2 in a Total Column

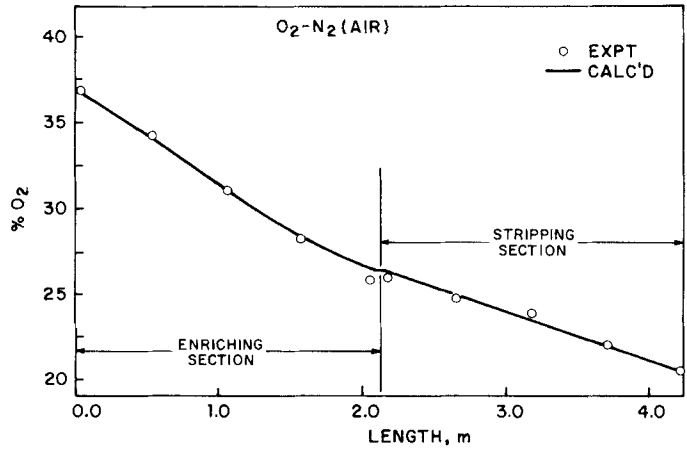

FIGURE 5 Shell-Side Composition Profile for O_2-N_2 in a Total Column

O_2 and 84.9% N_2 at 0.0400 and 0.0972 std cc/sec, respectively. The compressor load was 0.284 std cc/sec. The axial pressure loss was negligible again. It is interesting to compare this result with the conventional permeator result, which gives 28% O_2 at comparable operating conditions. Thus, this system clearly demonstrates that a continuous membrane column can separate a binary mixture beyond the maximum limit obtainable by a single stage conventional permeator.

Tube-side sampling was impossible; however, the calculated simulation profiles of composition and flow rate are shown in Fig. 6. The values for terminal points agree very well with experimentally measured values.

3. Performance Characterization

Using a concept similar to that of characterizing the packed columns of distillation, extraction, and gas absorption, the number of membrane units (NMU) and the height of a membrane

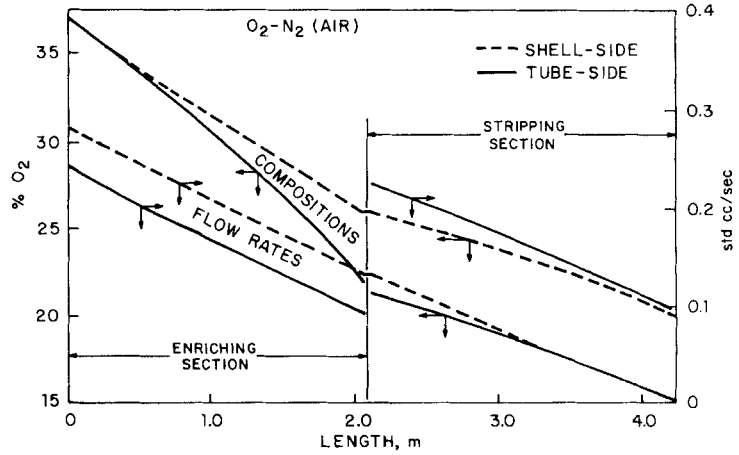

FIGURE 6 Calculated Profiles of Compositions and Flow Rates for O_2-N_2 in a Total Column

unit (HMU) were introduced (26) in place of NTU and HTU, respectively. The definitions are:

$$NMU \equiv \int_B^T \frac{dx}{x-yP_r-x\{(1-Q_r)(x-yP_r) + Q_r(1-P_r)\}} \tag{1}$$

and

$$HMU \equiv \frac{Z}{\int_B^T \frac{2\pi NQ_1P_i}{q\ \ln(r_o/r_i)}\, dz} \tag{2}$$

Then the total height of the continuous membrane column becomes:

$$Z = (NMU)(HMU) \tag{3}$$

It was also shown that Eq. (2) can be approximated as

$$HMU \simeq \frac{(q_T - q_B)\ \ln(r_o/r_i)}{2\pi NQ_1P_i \ln(q_T/q_B)} \tag{4}$$

when axial pressure loss is negligible. At total reflux, x equals y at every point in the column, and Eq. (1) simplifies to:

$$NMU = \int_B^T \frac{dx}{(1-Q_r)(1-P_r)x(1-x)} = \frac{1}{(1-Q_r)(1-P_r)} \ln\left(\frac{x_T}{x_B}\right)\left(\frac{1-x_B}{1-x_T}\right) \tag{5}$$

Substituting Eqs. (4) and (5) into Eq. (3), yields:

$$Z = (NMU)(HMU) \simeq \frac{(q_T-q_B)\ln(r_o/r_i)\ \ln[x_T(1-x_B)/x_B(1-x_T)]}{2\pi NQ_1P_i(1-Q_r)(1-P_r)\ \ln(q_T/q_B)} \tag{6}$$

This equation may be used in design.

It was also shown that Eq. (1) can be integrated to yield:

$$NMU = \int_B^T \frac{dx}{(1-Q_r)x(1-x)} = \frac{1}{(1-Q_r)} \ln\left(\frac{x_T}{x_B}\right)\left(\frac{1-x_B}{1-x_T}\right) \tag{7}$$

when the pressure ratio becomes zero but not necessarily at a total reflux condition (product may be withdrawn). Combining this equation and Eq. (4) into Eq. (3) results in:

$$Z \simeq \frac{(q_T-q_B)\ln(r_o/r_i)}{2\pi NQ_1P_i(1-Q_r)} \frac{\ln[x_T(1-x_B)/x_B(1-x_T)]}{\ln(q_T/q_B)} \tag{8}$$

Another useful equation was derived relating the flow rates to the compositions at the top and bottom of a continuous membrane column for cases of either $P_r = 0$ or total reflux:

$$\left(\frac{q_T}{q_B}\right)^{\alpha-1} = \left(\frac{x_T}{x_B}\right)\left(\frac{1-x_B}{1-x_T}\right)^{\alpha} \tag{9}$$

This equation is exact and involves only the terminal point conditions.

COMPARISON OF CONTINUOUS MEMBRANE COLUMN WITH CONVENTIONAL PERMEATOR

There are many ways to compare the performance of a continuous membrane column with that of a conventional gas permeator. As mentioned previously, there is no limit of enrichment attainable in a given continuous membrane column whereas there is a definite upper limit in a single stage conventional permeator. While this fact is the most important feature of a continuous membrane column, several quantitative comparisons of various operating parameters are also of interest. For the purpose of illustration, the system of O_2 - N_2 (air) with

silicone rubber as the membrane was chosen. For this system, $\alpha = 2.018$ and the maximum enrichment attainable in a conventional permeator is 34.78% O_2.

The amount of oxygen enriched product per unit feed varies as a function of product composition. For a conventional permeator, the permeate composition vs. the stage cut, which is the ratio of product to feed, gives this relationship. Based on the countercurrent plug flow model as shown in Fig. 7, the product composition can be calculated as a function of stage cut using the material balance and Eq. (9) where $q_T = F$, $q_B = R$, $x_T = x_F$, and $x_B = x_R$ are substituted.

$$x_F F = y_P P + x_R R \tag{10}$$

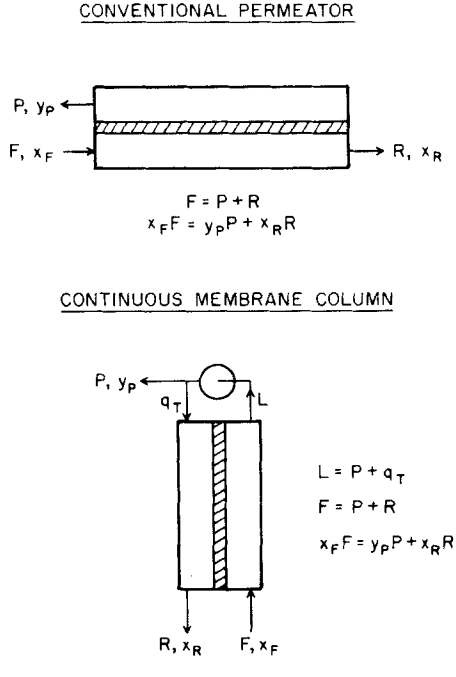

FIGURE 7 Schematic Flow Diagrams

$$\left(\frac{F}{R}\right)^{\alpha-1} = \left(\frac{1-x_R}{1-x_F}\right)^{\alpha}\left(\frac{x_F}{x_R}\right) \tag{11}$$

The two unknown quantities x_R and y_P can be solved from these equations and the results are plotted in Fig. 8 together with the results obtained for a continuous membrane column.

For a continuous membrane column there exists no one-to-one correspondence between the product composition and the product to feed ratio in general. At a given product concentration, the product flow rate can be varied by changing the compressor load. The product flow rate can always be set at zero for the lowest value, which gives the total reflux condition. However, it can not be increased indefinitely in order to produce a specified product composition. In fact, there exists an optimum condition in which the maximum product per unit compressor load can be achieved. Referring to Fig. 7, the material balance around the compressor gives

$$\frac{P}{L} = 1 - \frac{q_T}{L} = 1 - \frac{q_T}{q_T + F - R} = 1 - \frac{q_r}{q_f + F_r - 1} \tag{12}$$

where $q_r = q_T/R$ and $F_r = F/R$. The product per unit compressor load can be maximized by differentiating with respect to F_r and setting the result equal to zero:

$$\frac{d(P/L)}{dF_r} = 0 \tag{13}$$

which results in the following equation

$$(F_r - 1)\frac{dq_r}{dF_r} = q_r \tag{14}$$

The derivative $\frac{dq_r}{dF_r}$ can be computed from Eq. (9) after substituting

$q_B = R$, $x_T = y_P$, and $x_B = x_R$ and using the material balance equations to eliminate x_R as shown below:

$$(q_r)^{\alpha-1} = \left(\frac{y_P}{x_R}\right)\left(\frac{1-x_R}{1-y_P}\right)^{\alpha} = \left[\frac{y_P}{(x_F-y_P)\ F_r + y_P}\right]\left[\frac{1-(x_F-y_P)F_r-y_P}{1-y_P}\right]^{\alpha} \quad (15)$$

Then, solving Eq. (14) for F_r, the optimum F_r value is obtained as follows:

$$F_r = \frac{(\alpha-1)\ x_F(1-x_F)}{(x_F-y_P)\{(\alpha-1)x_F - \alpha\}} + 1 \quad (16)$$

Once the optimum F_r is obtained, the corresponding x_R value can be calculated from the material balance:

$$x_R = (x_F-y_P)F_r + y_P = \frac{x_F}{\alpha(1-x_F) + x_F} \quad (17)$$

Substituting this into Eq. (15), the value of q_r is known. Finally, substituting the values of q_r and F_r into Eq. (12) gives the maximum P/L ratio for a given product concentration. From a material balance, P/F can be easily calculated as follows:

$$\frac{P}{F} = 1 - \frac{1}{F_r} \quad (18)$$

This optimum value is plotted as a function of product concentration in Fig. 8. In the lower range of product concentration, there is not much difference between the amounts of product generated by a conventional permeator and by a continuous membrane column. However, as the product concentration increases, the amount of product given by a conventional cell decreases rapidly while that by a continuous membrane column decreases more grad-

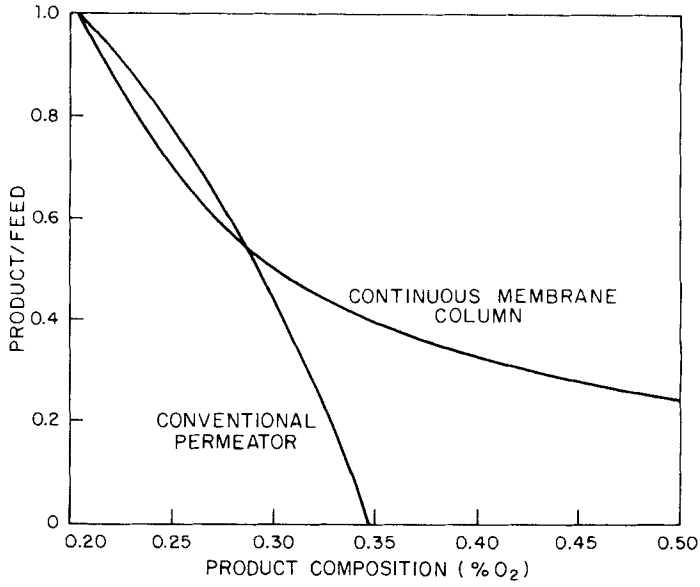

FIGURE 8 Comparison of Product Rates

ually. At an oxygen concentration of 34.8%, the P/F ratio for a conventional cell becomes zero and beyond that point it is impossible to enrich O_2 with a single stage. On the other hand, the continuous membrane column may still be operated with a finite amount of product stream withdrawn as illustrated in Fig. 8.

Another relevant quantity in gas permeator operation is the compressor load. The major operating cost is frequently due to the compressor load. For a conventional permeator, the inverse of P/F is directly the compressor load. However, for a continuous membrane column, the inverse of P/L, which was described above, gives the compressor load instead. In Fig. 9, the compressor load per unit product is plotted against product composition for both cases. It should be noted that the conventional permeator requires less compressor load for most of the concentration range below the maximum limit as expected. Near the limiting value of enrichment concentration, the two curves cross over and

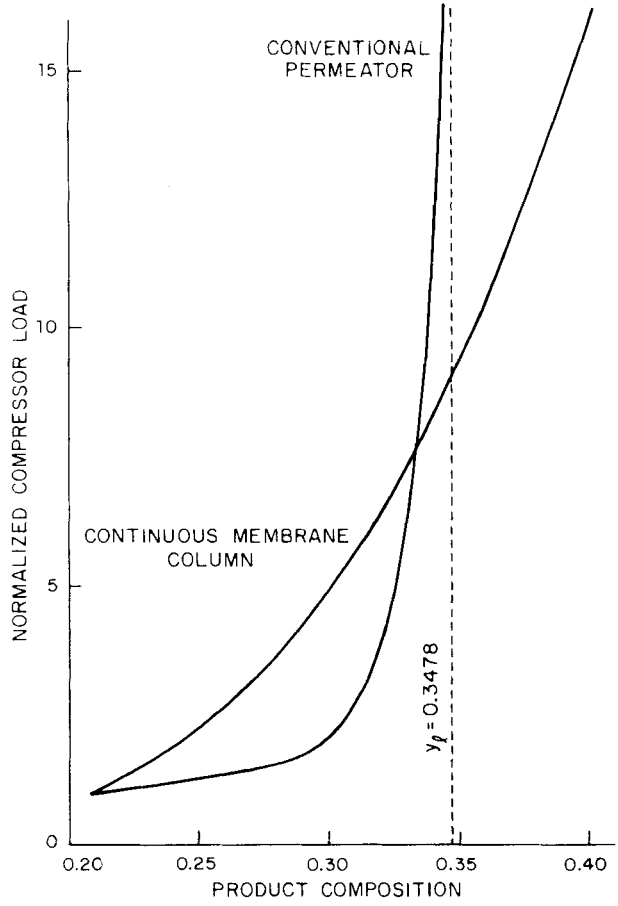

FIGURE 9 Comparison of Compressor Loads

the conventional compressor load increases much more sharply than that of a continuous membrane column. It is obvious that the continuous membrane column is more advantageous in higher concentration because it is a continuous cascade.

Finally, the membrane area requirements are compared. Instead of calculating the actual membrane areas, the ratio of membrane areas is presented in Fig. 10 for the purpose of compari-

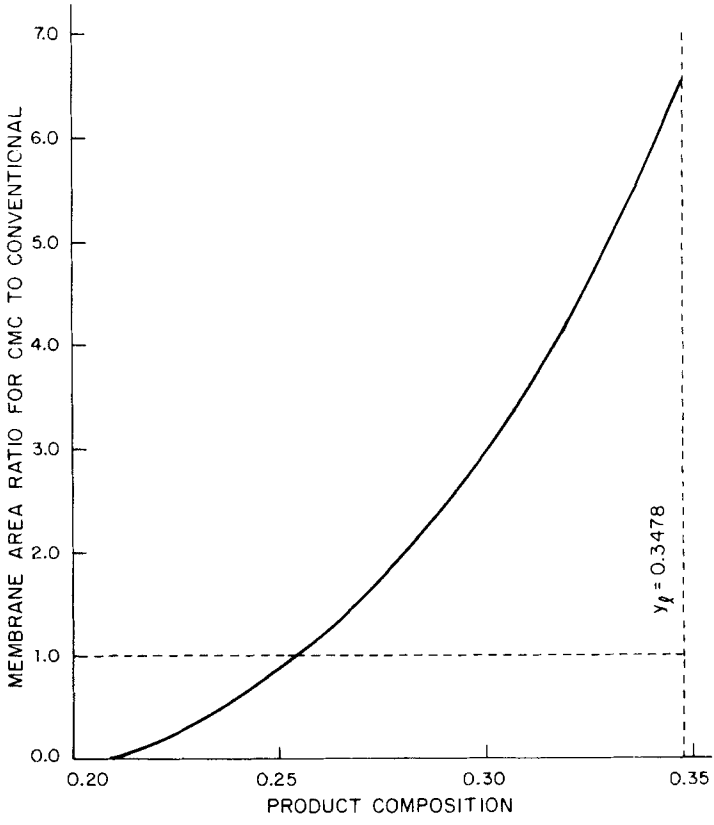

FIGURE 10 Comparison of Membrane Areas

son. The membrane areas are porportional to the permeate rates. For a conventional permeator, the membrane area is:

$$S_C = \frac{P\ t}{(P_i - P_o)\ \overline{Q}_C} \tag{19}$$

and similarly, for a continuous membrane column, the membrane area is:

$$S_M = \frac{(L - F)\, t}{(P_i - P_o)\, \bar{Q}_M} \tag{20}$$

The two units are compared at identical operating conditions. Therefore, it is assumed that the average permeabilities are the same. Dividing Eq. (20) by Eq. (19) yields:

$$\frac{S_M}{S_C} = \frac{L}{P} - \frac{F}{P} \tag{21}$$

Since the product rates are identical for both, the right hand side of Eq. (21) may be interpreted as the difference of normalized compressor load and feed rate for the continuous membrane column. This quantity has already been calculated above. It is plotted against product composition in Fig. 10.

It is interesting to note that for the low range of product concentration, the continuous membrane column requires less membrane area than the conventional permeator. At about 25% O_2, the membrane requirements become identical for both cases, above which the continuous membrane column needs more membrane area than the conventional unit. The comparison is possible only for the concentration range below the limiting value (34.78% O_2).

In overall comparison, an economic study must be carried out, which is beyond the scope of the present paper. However, a qualitative conclusion may be drawn by inspecting Fig. 9 and 10. Depending on the relative costs of membrane and power, there may be a region of concentration in which the conventional permeator is more economical than the continuous membrane column. However, near the limiting concentration it appears that the continuous membrane column would be better than the conventional one. Of course, when the desired product concentration is greater than the limiting value, the continuous membrane column is the only choice, unless the conventional units are cascaded.

CONCLUSIONS

The continuous membrane column provides a new separation technique. Higher enrichments, exceeding the conventional limit, have been achieved experimentally without cascading individual units. The comparisons of compressor load and membrane area requirements indicate that the continuous membrane column is not needed when the enriched product is of low concentration. However, for higher enrichment, the continuous membrane column can replace the conventional multistage cascade.

ACKNOWLEDGMENT

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SYMBOLS

F = feed flow rate, std cc/sec

F_r = F/R

HMU = height of a membrane unit as defined by Eq. (2), cm

L = compressor load, std cc/sec

N = number of capillaries

n = number of stages

NMU = number of membrane units as defined by Eq. (1)

P = top product flow rate, std cc/sec

P_i = absolute local tube-side pressure, cm Hg

P_o = atmospheric pressure, cm Hg

P_r = P_o/P_i

q = tube-side flow rate, std cc/sec

q_r = q_T/R

Q = permeability coefficient, $\frac{(\text{std cc})\ (\text{cm})}{(\text{sec})(\text{cm}^2)\ (\text{cm Hg})}$

$\bar{Q}$ = average permeability, $\frac{(\text{std cc})\ (\text{cm})}{(\text{sec})(\text{cm}^2)\ (\text{cm Hg})}$

Q_r = Q_2/Q_1

r = radius of capillary, cm

R = bottom product flow rate, std cc/sec

S = membrane area, cm^2

t = membrane thickness, cm

x = mole fraction of the more permeable component in tube-side

y = mole fraction of the more permeable component in shell-side

z = axial coordinate, cm

Z = total column height, cm

Greek Letters

α = separation factor (Q_1/Q_2)

π = 3.141 592 ...

Subscripts

1 the more permeable component

2 the less permeable component

B at the bottom of the column

C conventional permeator

F feed

i inside

ℓ limiting value

M continuous membrane column

o outside

P top product

R bottom product

r ratio

T at the top of a column

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