Theoretical Analysis of Butane Isomers Separation using Various Membrane Process Configurations

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Abstract: The main purpose of this study is performance investigation of single stage membrane, continuous membrane column (CMC) and countercurrent recycle membrane cascade (CRC)during butane isomers separation in the typical LPG plant. As a case study, the required total membrane area and compressor duty for three type configurations for separation of butane isomers produced in LPG unit of Tabriz refinery was evaluated. For this purpose, a typical H-ZSM-5 zeolite membrane with n-butane/isobutene selectivity around 10 was considered. The optimum scheme was then selected by means of minimum total membrane area and compressor duty requirements. Moreover, the effects of key parameters such as selectivity, feed to permeate pressure ratio and composition of the permeate stream on the membrane surface area was evaluated.

Keywords: Gas separation, Membrane process, Modeling, Continuous membrane column, Countercurrent recycle cascade.

1. INTRODUCTION

Membrane technology for separation purposes are becoming much more interesting field in comparison with the other conventional separation techniques such as distillation, absorption, extraction, etc.[1, 2]. Modeling of this process is a vital step in developing and analyzing their performance in both pilot and industrial scale. The first design model for membrane permeators was presented by Weller and Steiner in 1950 [3]. They derived an analytical solution based on Fick's law, assuming negligible pressure drop and cross-flow along the permeate side of the membrane. The Most studies for gas separation processes using membranes have employed a single stage mode [4, 5]. Nonlinear ordinary differential equations that govern the performance of the membrane modules for gas separations are well known and have been solved by many investigators [4-10]. Shindo *et al*. [5] developed approximate calculation methods for multi component separations in five flow patterns: one-side mixing, complete mixing, cross flow, countercurrent and concurrent flow. Krovvidi *et al*. [8] tried to solve the governing differential equations directly as a two-point boundary value problem using shooting technique. Aghaeinejad-Meybodi *et al*. [10] presented the corrected operating line method (OLM) for modeling of binary gas mixtures separation in membranes. Kaldis *et al*. used the orthogonal collocation technique to solve the mathematical model which describes the binary gas mixture separation in hollow fiber asymmetric membrane [11, 12]. Razmjoo and Babaluo used asymptotic analysis of binary gas mixture separation by nanometric tubular ceramic membranes for cocurrent and countercurrent Flow Patterns [13]. They also presented a combinational approach (combination of asymptotic and approximate methods (CAAM)) to simulate the separation of binary gas mixtures in nanometric tubular ceramic membranes with co-current and countercurrent flow patterns [14, 15]. An alternative way to achieve high product purity and high recovery of the desired species is to design a suitable membrane cascade. Multistage membrane configurations and membrane arrangements similar to the distillation column have been considered to meet industrial requirements for high purity products [16]. One of the early studies on the characteristics of multistage membrane cascades was made by Pan and Habgood [17]. They considered both ideal and non-ideal cascades with up to six membrane stages. Stern *et al*. [18] studied the behavior of single stage membrane with permeate recycle, and continuous membrane columns (CMC). Avgidou *et al*. [16] studied membrane cascade schemes for the separation of LPG olefins and paraffins. The design of optimum membrane cascade schemes (CMC and CRC

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configurations) for olefin/paraffin separations has not been discussed in any detail in the open literature.

The present work examines the performance of membrane cascade schemes in gas separation with cross flow pattern. These include: single-stage permeation, two- type of continuous membrane column (CMC) and countercurrent recycle cascade (CRC). For each case, the required membrane area and compressor duty were obtained. The optimum scheme was then selected by means of minimum total membrane area and compressor duty requirements. Also the effects of key parameters such as selectivity, ratio of feed to permeate pressure ratio and composition of the permeate stream on the membrane surface area was evaluated.

2. THEORY DESCRIPTION

2.1. Single Stage Membrane with Cross Flow Pattern

A cross flow ideal membrane stage shown in Figure **1**. In this stage it is assumed that the gas on the high pressure side of the membrane flows parallel to the membrane with no mixing, and that the membrane is situated sufficiently far away from the exiting permeate stream so that the permeate flows perpendicular to, and away from, the membrane, again with no mixing taking place near the permeate side of the membrane. With these assumptions, the permeate composition at any point near the membrane is determined by the relative rates of permeation of the high pressure gas mixture at that point. The exiting permeate composition from this stage is the pooled average permeate composition ($\bar{y}$) [19].

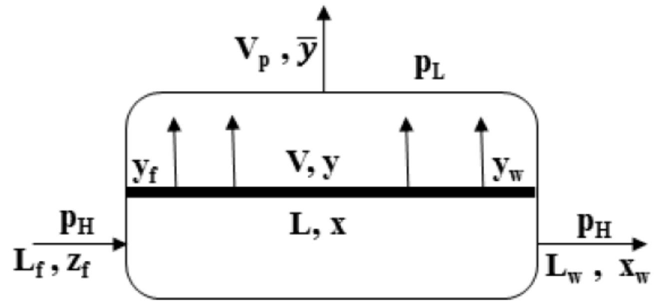

Figure 1: Schematic of a general cross flow pattern.

2.2. Continuous Membrane Column (CMC)

The continuous membrane column (CMC) was introduced by Hwang and Thorman in 1980 [20]. The continuous membrane cascade with cross flow pattern is shown in Figure **2**. As shown in this Figure, the column is divided into an enriching and a stripping section, by the point of the feed inlet. As the mixture travels downwards to the high-pressure side, it is depleted of the more permeable components, which preferentially permeate through the membrane into the low-pressure side. In CMC (1) scheme (Figure **2a**) the

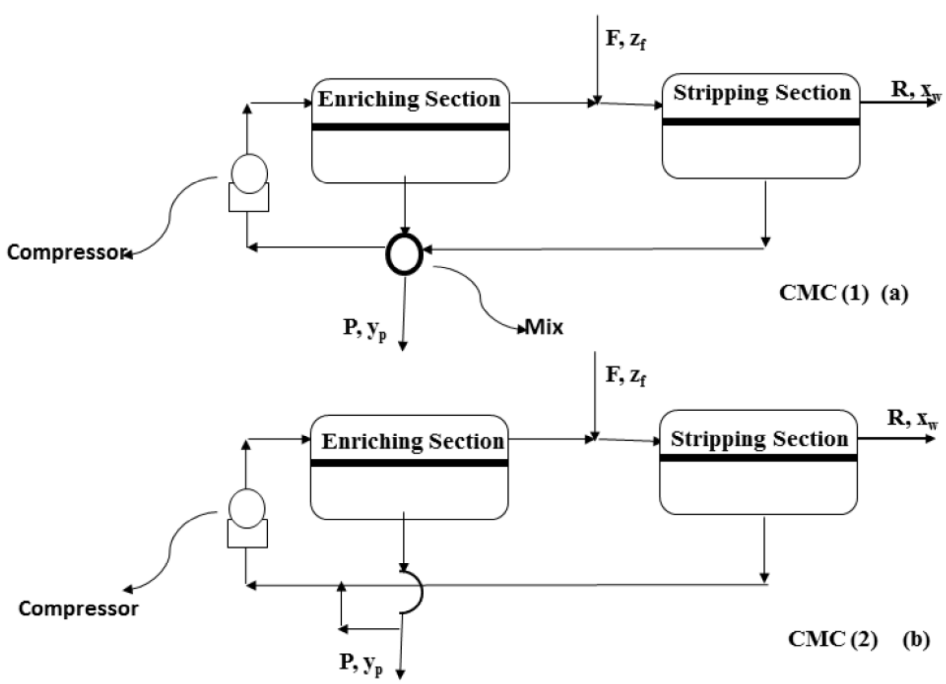

Figure 2: Two- type continuous membrane column configurations with cross flow pattern.

entire permeate streams from enriching and stripping sections are mixed and a fraction of this stream is compressed and used as feed in enriching section. In CMC (2) scheme (Figure **2b**) all of the permeate stream from the striping section and a fraction of the enriching section are compressed and set as feed to the enriching section. Therefore, a binary mixture can be theoretically separated into two components of any desired degree of purity in a CMC operated at a sufficiently high reflux ratio and low stage cut, regardless of the membrane selectivity and the used membrane area [16].

2.3. Countercurrent Recycle Cascade (CRC)

Many separations are carried out in countercurrent recycle cascades such as shown in Figure **3**. The countercurrent recycle cascade is a multistage design scheme where separates a high pressure binary feed stream, rate F, composition z_f, into an enriched product stream, rate P, composition y_p, and a depleted bottom stream, rate R, composition x_w. In this cascade, the intermediate side streams are recycled. Therefore, the CRC mode achieves significantly higher yields compared to the non-recycle cascades. A CRC is divided into two sections, the enriching one and the stripping one. The feed is introduced between the enriching and the stripping sections. In analogy to conventional stage operations (*e.g*. distillation), a fraction of the feed travels upwards through the enriching section and it is enriched in the more permeable components, the rest feed travels downwards through the stripping section and it is depleted of the more permeable components[21]. The recycle flow pattern is characteristic of a general countercurrent recycle cascade (CRC), that is, the tails stream from stage i+1 and the heads stream from stage i-1 combined make up the feed to a general stage i [18].

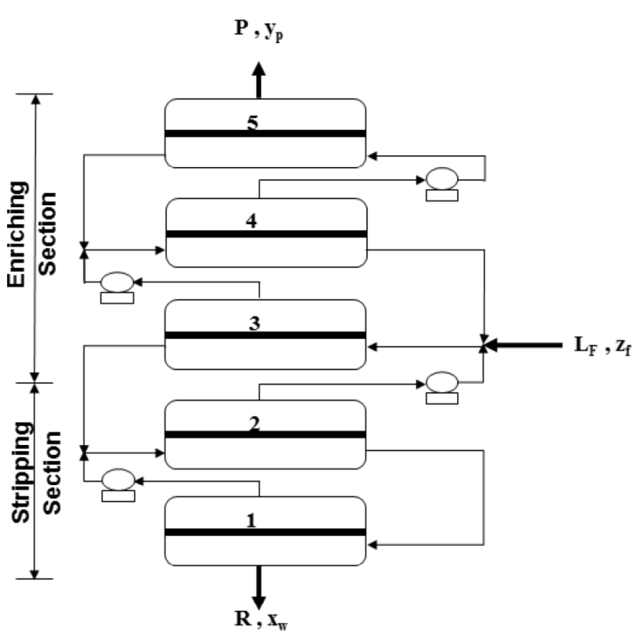

Figure 3: Countercurrent recycle cascade composed of membrane stages.

2.4. Model Development

The main assumptions are:

- The feed is a binary gas mixture and there is no purge on the permeate side.
- The permeability coefficient of each component is that of the pure component.
- Negligible pressure drop is experienced by feed and permeate streams.

In this study, cross flow pattern will be assumed in modeling the single stage and different multistage membrane cascades.

For a binary gas mixture, mass conservation over the differential membrane area (dA) in a single stage (Figure **1**), leads to [22]:

$$y_i dV = \frac{P_i}{\delta}(p_H x_i - p_L y_i)dA \qquad (i = 1,2) \tag{1}$$

Where y_i is the local permeate concentration.

The average permeate concentration from feed inlet to any point over the membrane is shown by $\bar{y}$.

Manipulation of Eq. (1) leads to the relationship among the components:

$$\frac{P_1}{y_1}(p_H x_1 - p_L y_1) = \frac{P_2}{y_2}(p_H x_2 - p_L y_2) \tag{2}$$

Substitution p_H/p_L = r and P_1/P_2 =α in Eq. (2) gives:

$$\alpha y_2(r x_1 - y_1) = y_1(r x_2 - y_2) \tag{3}$$

x_i and y_i (i=1,2) may be eliminated through the definition of mole fractions, $\sum y_i = 1.0$ and $\sum x_i = 1.0$. Substitution of y_2=1-y_1 and x_2=1-x_1 into Eq. (3) lead to:

$$(\alpha-1)y_1^2 - (\alpha-1)y_1 - ry - (\alpha-1)rx_1y_1 + \alpha r x_1 = 0 \tag{4}$$

Substitution λ=α/(α-1) in Eq. (4) gives:

$$y_1^2 - y_1 - r(\lambda - 1)y_1 - rx_1y_1 + \lambda rx_1 = 0 \quad (5)$$

Then

$$\frac{rx_1}{y_1} = \frac{1 + r(\lambda - 1)}{\lambda - y_1} \quad (6)$$

Rearrangement of Eq. (1) and using r= p_H/p_L results in:

$$\frac{p_L P_1}{\delta} dA = \frac{dV}{\frac{rx_1}{y_1} - 1} \quad (7)$$

Substitution of Eq. (6) into Eq. (7) leads to:

$$\frac{p_L P_1 dA}{\delta} = \frac{\lambda - y_1}{(r-1)(\lambda - 1)} dV \quad (8)$$

By integration between the limits 0 to A and 0 to V_p, we have:

$$\frac{p_L P_1 (r-1)(\lambda - 1)A}{\delta} = V\left[\lambda - {}^1\!/_V \int_0^{V_p} y_1 dV\right] \quad (9)$$

The expressions (${}^1\!/_V \int_0^{V_p} y_1 dV$), are equal to the pooled average of the corresponding mole fraction in the permeate stream ($\bar{y}$)[22].

$\bar{y}$ is substituted in Eq. 9 and with rearrangement, for binary gas mixture, an explicit expression is obtained for the calculation of membrane area.

$$A = \frac{\theta L_f (\lambda - \bar{y})\delta}{p_L P_1 (r-1)(\lambda - 1)} \quad (10)$$

where θ is stage cut and defined as the ratio of permeate to feed rate.

Flow rate and composition of permeate for a two component system can be obtained from an expression derived by integration of the differential equation, representing the mass balance on a differential increment:

$$-\frac{dV}{F - V} = \frac{dx_1}{y_1 - x_1} \quad (11)$$

Both x_1, and dx_1, can be substituted with functions of y_1 and dy_1, obtained from the two-component form of Eq. (5). Integration of the resulting expression between the limits 0 to V_p and y_f to y_w and substitution θ = V_p/F, yields:

$$\theta = 1 - \left(\frac{\lambda - y_w}{\lambda - y_f}\right)\left(\frac{1 - y_w}{1 - y_f}\right)^a \left(\frac{y_w}{y_f}\right)^b \quad (12)$$

Where

$$a = \frac{1 - \lambda r}{r - 1} \qquad b = \frac{\lambda r}{r - 1} - 1$$

The boundary values at the feed entrance of the system (y_f) and residue exit (y_w) are obtained from Eq. (5) with x_f and x_w, in turn. The value of average composition of permeate stream is calculated by Eq. (13):

$$\bar{y} = \frac{x_f - x_w}{\theta} + x_w \quad (13)$$

In modeling the multistage membrane cascades, cross flow pattern equations for every stage and no-mix conditions are used, where the no-mix criteria are as assigned below:

For multistage CRC schemes, the compositions of head and tail streams forming the feed of every stage are the same. For CMC schemes, at the feed point, the compositions of streams are equal to the feed composition [16].

The compressor duty for each scheme is calculated as:

C.D (mol/Pa.s^{-1}) = (sum of all streams flow rates to various compressors) × (p_H - p_L) (14)

The feed stage is taken as the initiation point for calculation and the calculations are continued until the enriching and striping sections reach the desired concentrations.

3. MODELING CONDITIONS

The objective of this case study is to evaluate membrane process for the separation of nC_4 and iC_4 gas mixture. Table **1** illustrates the isobutene rich gas stream specification of Tabriz refinery LPG unit. For simplicity of this case study compositions of C_3, nC_5 and iC_5 have been neglected and their quantities are assigned as nC_4 and iC_4 respectively. Table **2** shows specification of the feed stream used in the modeling.

Table1: Specification of Gas Stream of Tabriz Refinery LPG Unit

Components	Mole Percent
C_3	0.5
iC_4	36.6
nC_4	58.8
iC_5	3.1
nC_5	1
Total	100
Operating Conditions	
Temperature (^0C)	65
Pressure (Kg/cm^2)	12-15
Rate (Ton/hr)	13.2

Table 2: Specification of the Feed Stream used in the Modeling

Components	Mole Percent
iC_4	39.7
nC_4	60.3

In this investigation H-ZSM-5 zeolite membrane has been assumed for modeling the membrane separation process of butane isomers. The selectivity of 10 for nC_4 to iC_4 gas mixture and permeability coefficient of $3.3 \times 10^{-9}\ \mathrm{mol.m/(Pa.m^2.s)}$ for nC_4 was considered based on the data published by Gardner *et al.* [23].

4. RESULT AND DISCUSSION

4.1. Model Validation

In order to validate the mathematical model, simulation results has been compared with Seda *et al*. [24] experimental data. As shown in Figure **4**, a good agreement was achieved between our model results and experimental data of Seda *et al*. for stage cut values less than 0.6, in which the maximum error is 0.75%, while the calculated maximum error for higher values of stage cut (>0.6) is 5%.

4.2. Evaluation of Membrane Schemes

As mentioned earlier, an optimum design is minimizing the total membrane area and compressor duty with a highly pure product. Evaluations of three different membrane schemes have been considered in this study. These schemes are:

1. Single stage membrane with a cross flow pattern.

2. Continuous membrane column (CMC).

3. Countercurrent recycle membrane cascade (CRC).

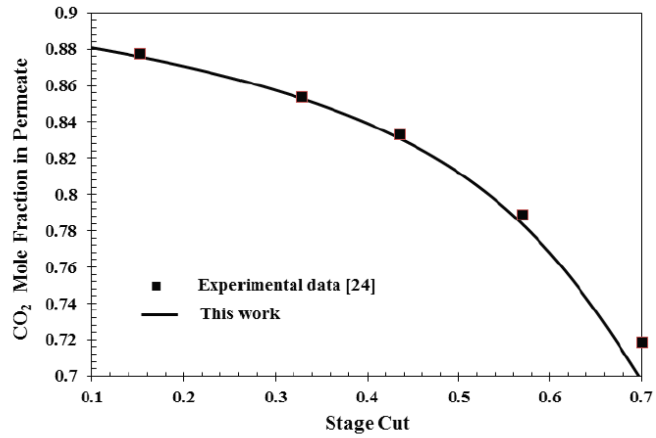

Figure 4: The comparison of modeling results and experimental data [24].

For this mean, for each schemes, the total surface area of the membranes and compressor duty are calculated for the same separation using the discussed model.

For a single stage system with a cross flow pattern, the model has been used thou nC_4 reaches 0.0482 in the residue stream.

Table **3** shows the results of the modeling for a single stage membrane system with a cross flow pattern.

As shown in Table **3**, highly purity nC_4can't be obtained in the permeate stream using a single stage membrane with a cross flow pattern making this system an inappropriate membrane system and the manipulation should be assigned using multistage membrane cascades. Highly pure nC_4 and iC_4 products can be obtained using CRC and CMC schemes.

Table 3: Results of Modeling for Single Stag Membrane with Cross Flow Pattern

x_f	x_w	y_p	Stage Cut	L_f (mol/s)	P (mol/s)	R (mol/s)	Membrane Area (m^2)
0.603	0.0482	0.7852	0.7527	63.2	47.57	15.63	37938.4

Compositions and stream flow rates of nC_4 for the feed, residue and permeate streams for the CMC (1), CMC (2) and CRC schemes are shown in Figures **5** and **6** respectively. Tables **4** and **5**, show the simulation results using CMC and CRC schemes. As it can be clearly seen the concentration of the nC_4 in the permeate and residue streamers are y_p=0.9944 and x_w=0.0482 respectively using CMC and CRC schemes.

As shown in Tables **4** and **5**, the CMC(1) and CMC(2) schemesare the less efficient for the specified separation, requiring about 17.5 and 6.5 times more

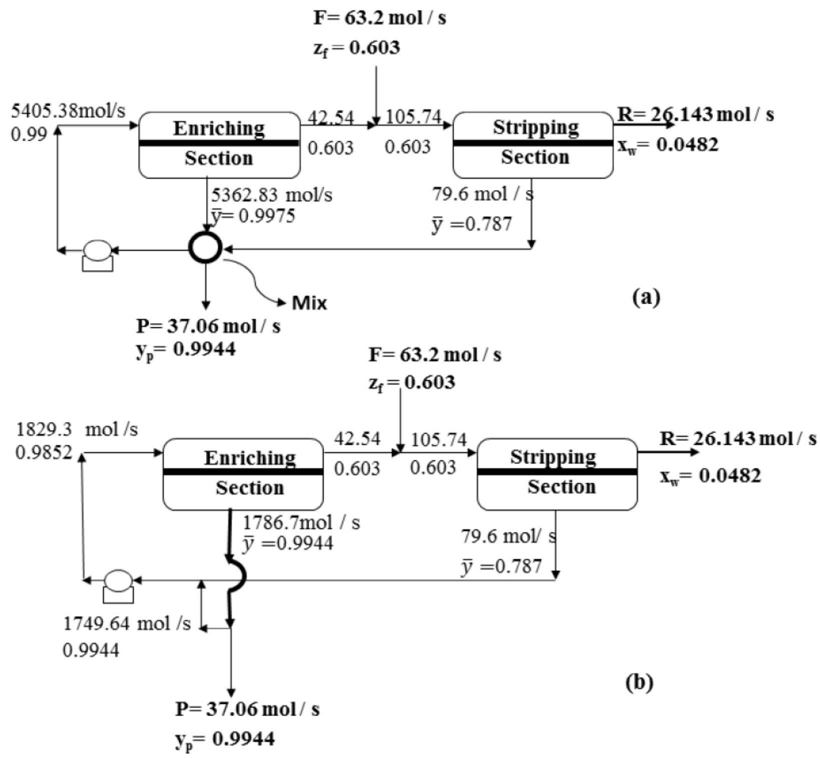

Figure 5: Compositions and stream flow rates of nC4 calculated for the **a**) CMC(1) and **b**) CMC(2)schemes.

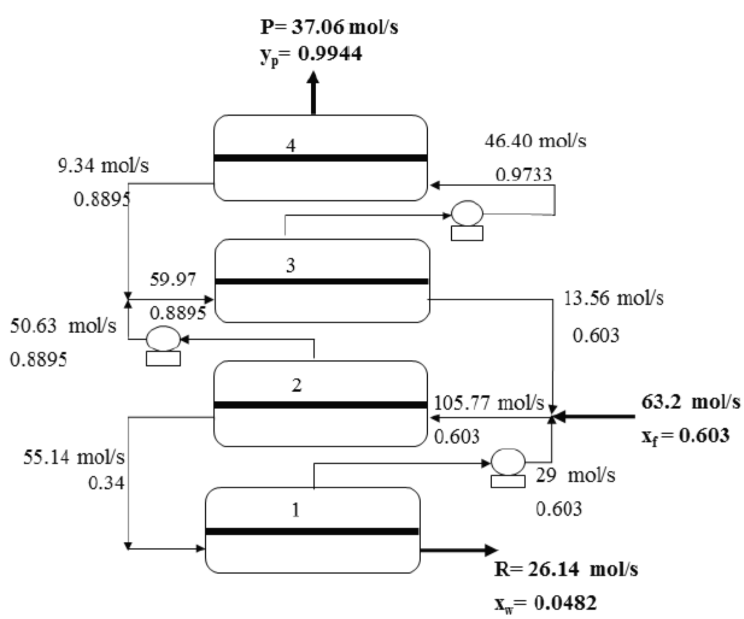

Figure 6: Compositions and stream flow rates of nC4 calculated for the CRC scheme.

Table 4: Simulation Results for CMC Scheme

	Membrane Area (m^2)			Stage Cut		Compressor Duty ($mol/atm.s^{-1}$)
	Stripping Sec.	Enriching Sec.	Total	Stripping Sec.	Enriching Sec.	
CMC(1)	63476.50	1490788.57	1554265.06	0.7527	0.9921	60247.04
CMC(2)	63476.50	510272.38	573748.88	0.7527	0.9767	20122.53

Table 5: Simulation Results for CRC Scheme

Stage	Membrane Area (m^2)	Stage Cut	Total Membrane Area (m^2)	Compressor Duty ($mol/atm.s^{-1}$)
1	36045.3524	0.5259	89732.45	1386.24
2	27450.3888	0.4786		
3	15651.3258	0.7738		
4	10585.3862	0.7987		

membrane area and 43.5 and 14.5 times more compressor duty respectively, than the CRC scheme. Therefore, parametric studies have been presented in the next section for the CRC scheme.

4.3. Parametric Evaluation of CRC Performance

4.3.1. Final Permeate Composition

The effect of final permeate composition on the total membrane area and compressor duty in CRC scheme to obtain final residue composition of x_w=0.05 is shown in Figure **7**. As can be clearly seen from this figure, by increasing the final composition of permeate, the total membrane area and compressor duty increase dramatically at y_p> 0.99.

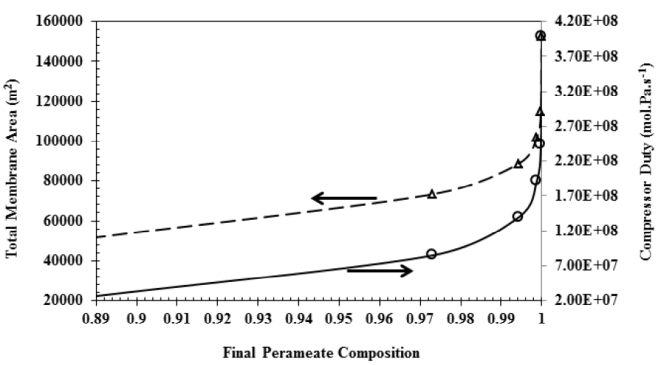

Figure 7: Effect of final permeate composition on total membrane area and compressor duty in CRC scheme (z_f = 0.603, x_w= 0.05, α=10, r=12).

The effect of final permeate composition on the number of required stages to obtain final residue composition of x_w=0.05 is shown in Figure **8**. As it is clearly illustrated in Figure **8**, the number of stages increases tremendously as the final permeate composition is greater than 0.99.

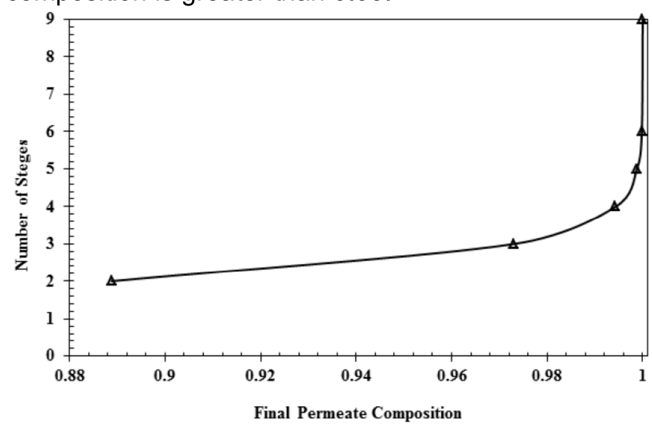

Figure 8: Effect of final permeate composition on number of stages in CRC scheme (z_f = 0.603, x_w= 0.05, α=10, r =12).

4.3.2. Ideal Selectivity

The changes in the trend of final permeate and residue composition with ideal selectivity when there are four membrane stages is shown in Figure **9**.

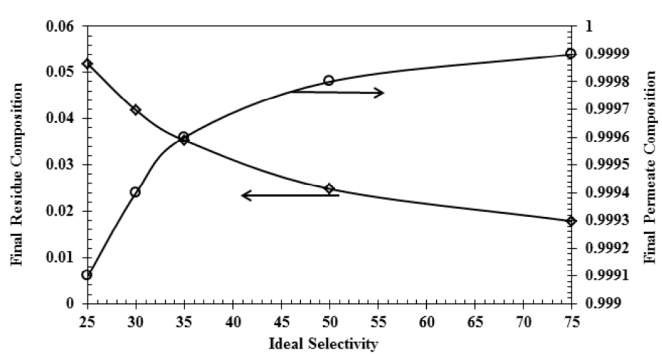

Figure 9: Effect of ideal selectivity on final compositions of permeate and residue streams in CRC scheme (z_f = 0.603, r =12, for four stage).

As the ideal selectivity is increased with respect to nC_4, the nC_4 composition in the final permeate and residue streams are increased and decreased respectively, suggesting highly pure products to be obtained with increase in selectivity.

The trend of the total membrane area and compressor duty variations with ideal selectivity, is shown in Figure **10** at a constant residue composition (x_w=0.05)

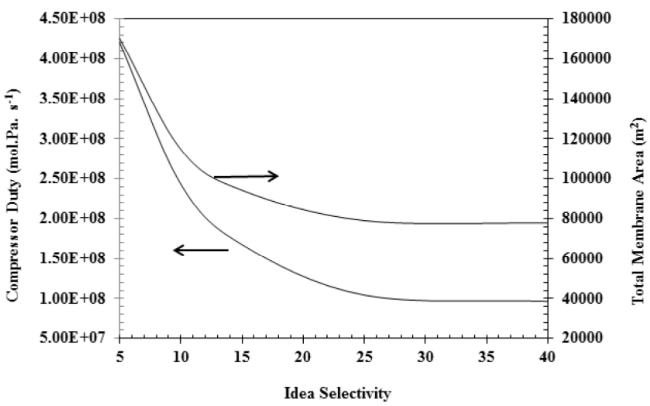

Figure 10: Effect of ideal selectivity on total membrane area and compressor duty in CRC scheme (z_f = 0.603, x_w= 0.05, r =12).

By increasing ideal selectivity, at first the total membrane area and compressor duty decrease dramatically and then tend to a plateau at high selectivity's (α >25).

4.3.3. Feed to Permeate Pressure Ratio (r)

The effect of ratio of feed to permeate pressure on the total membrane area and compressor duty is shown in Figure **11** at x_w=0.05.

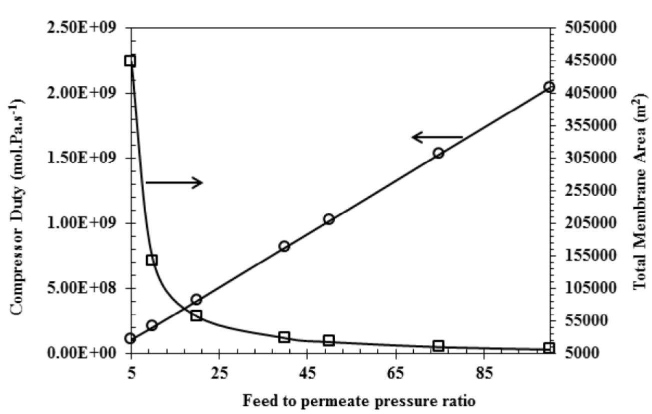

Figure 11: Effect of feed to permeate pressure ratio on total membrane area and compressor duty in CRC scheme (zf = 0.603, xw= 0.05, α=10).

A graphical representation confirms an optimum pressure ratio. As shown in this figure, the total membrane area decrease with increasing pressure ratio, while the compressor duty becomes lower when smaller pressure ratiois used. The optimum economic condition is located where the sum of membrane area cost and compressing cost becomes minimum.

5. CONCLUSION

In this work, three type single/multi stage membrane configurations were examined, *i.e*. single stage membrane, continuous membrane column (CMC) and countercurrent membrane cascade (CRC) assuming cross flow pattern in each stage. The simulation results show that highly purity nC_4 can't be obtained in the permeate stream using a single stage membrane with a cross flow pattern making this system an inappropriate membrane system and the manipulation should be assigned using multistage membrane cascades. Comparative studies between two multistage cascades showed that the CMC(1) and CMC(2) schemes are the less efficient for the specified separation, requiring about 17.5 and 6.5 times more membrane area and 43.5 and 14.5 times more compressor duty respectively, than the CRC scheme. Also parametric studies for the CRC schemes were investigated. With increasing the final composition of the permeate, the total membrane area and compressor duty increase dramatically at y_p> 0.99. With increasing selectivity, at first the total membrane area and compressor duty decrease dramatically and then tend to a plateau at high selectivities (α >25). Total membrane area decreases with increasing pressure ratio(r), while the compressor duty becomes lower when smaller pressure ratio is used. Therefore, it can be concluded that for economic and optimum design, the total cost of membrane area and compressing must be become minimum.

NOMENCLATURE

A	Membrane area (m^2)
a	Exponent in eqn. (12)
b	Exponent in eqn. (12)
F	Inlet feed flow rate (mol/ s)
L	Molar flow rate of the feed stream (mol/s)
pH	Feed side pressure (atm)
pL	Permeate side pressure (atm)
Pi	Permeability of component i (mol.m/m^2.s.atm),
P	Final permeate flow rate, (mol/s)

r	Feed/permeate pressure ratio, (p_H/p_L)
R	Residue flow rate (mol/s)
V	Molar flow rate of the permeate stream in the single stage (mol/s)
x	Mole fraction of components on the feed side
x_w	Mole fraction of more permeable component in residue stream
y	Mole fraction of components on the feed side
yp	Mole fraction of more permeable component in permeate stream
$\bar{y}$	Average permeate concentration in cross flow pattern
z_f	Mole fraction of more permeable component in feed stream

GREEK SYMBOLS

α	selectivity of the membrane for the more permeable component (P_1/P_2)
λ	Constant
θ	Stage cut
δ	Membrane thickness (m)

SUBSCRIPTS

f	Feed inlet
i	ith component
w	Residue end
p	Permeate outlet
1,2	Components 1 and 2 in binary gas mixture

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