Vol. 17, No. 3 (2018) 1083-1093
Revista Mexicana de Ingeniería Química
Simulación y Control

DESIGN OF A MEMBRANE PLANT FOR GAS SWEETENING BASED ON NEW POLYIMIDE MEMBRANES

DISEÑO DE UNA PLANTA DE MEMBRANA PARA ENDULZAMIENTO DE GAS BASADA EN NUEVAS MEMBRANAS DE POLIIMIDA

J.F. Palomeque-Santiago[1]*, J. Guzmán[1], D.J. Guzmán-Lucero[1], C. Jiménez-Martínez[2]

[1] *Instituto Mexicano del Petróleo. Eje Central Lázaro Cárdenas Norte No. 152, Col. San Bartolo Atepehuacan, México, D.F. C.P. 07730.*

[2] *Escuela Nacional de Ciencias Biológicas Zacatenco. Unidad Profesional Adolfo López Mateos. Av. Wilfrido Massieu Esq. Cda. Miguel Stampa s/n, Delegación Gustavo A. Madero, C.P.07738*

Received September 29, 2017; Accepted April 16, 2018

Abstract

A family of four new polyimides was synthesized with different CO_2 and CH_4 transport properties from fluorinated diaminotriphenylmethane and four different dianhydrides. The transport properties were used to simulate a gas membrane sweetening process in order to study their effect on the economy of the process. The membrane with the highest CO_2/CH_4 selectivity achieved the best performance for getting the product into specification, requiring less membrane area, obtaining higher methane recovery and lower gas processing cost (GPC). The effect of pressure feed was obtained by adding a second compressor to the feed stream; the results showed that the GPC exhibited an opposite trend to that of typical studies, increasing it with pressure; an explanation of this behavior is presented. The effect of feed flow on the GPC remained constant and presenting a linear behavior with membrane selectivity.

Keywords: natural gas sweetening, process simulation, polyimide membranes.

Resumen

Se sintetizó una nueva familia de poliimidas con diferentes propiedades de transporte de CO_2 y CH_4 a partir de diaminotrifenilmetano fluorado y 4 diferentes dianhídridos. Las propiedades de transporte se utilizaron para simular el proceso de endulzamiento de gas con el fin de estudiar sus efectos en la economía del proceso. La membrana con la mayor selectividad CO_2/CH_4 obtuvo un mayor desempeño para obtener un producto en especificación, requiriendo menor área de membrana, mayor recuperación de metano y menor costo de procesamiento de gas (CPG). El efecto de la presión se estudió adicionando un segundo compresor en la corriente de alimentación; los resultados mostraron que el CPG tuvo un efecto opuesto a estudios previos, incrementándose con la presión. Se presenta una explicación de este comportamiento. El efecto del flujo de alimentación en el CPG se mantuvo constante y presenta un comportamiento linear con la selectividad de la membrana.

Palabras clave: endulzamiento de gas natural, simulación de procesos, membranas de poliimida.

1 Introduction

The main advantages of the membrane separation processes are their low-energy consumption, low investment and maintenance costs and environmentally friendly nature. Permeability and selectivity are important properties, unfortunately, these properties move in opposite directions in membranes: as permeability increases, selectivity decreases and vice versa. Many polymeric materials have been investigated as potential membranes for gas separation but only some of them have found application in industrial gas plants (Baker and Lokhandwala, 2008; Yampolskii (2012); Zhang *et al*., 2013). Among the polymeric materials used as membranes for gas separation, polyimides (PIs) have been the subject of great interest because of their excellent physical properties, good chemical and thermal stabilities and improved processability, and offer excellent intrinsic CO_2/CH_4 separation properties (Wang *et al*., 2014; Plaza-Lozano *et al*., 2015; Sanders *et al*., 2013; Liaw *et al*., 2012; Qiu *et al*., 2013).

* *Corresponding author. E-mail*: jpalomeq@imp.mx
Tel. +52-55-9175 7398
doi: 10.24275/uam/izt/dcbi/revmexingquim/2018v17n3/Palomeque
issn-e: 2395-8472

In order to improve the gas separation properties of a polymer, bulky groups are introduced in the polymeric chain and at the same time the rigidity of the molecule is increased, resulting in a strong size sieving ability (Calle *et al.*, 2010). Fluorinated diaminotriphenylmethane is a diamine with bulky side groups, which produce an increase in both the fractional free volume (FFV) and rigidity, improving the gas permeation properties. The functional groups of the polymers contained in the anhydride moieties greatly affect the physical properties of the materials such as the packing density, chain rigidity, FFV, solubility in common dissolvents, and processability for forming membranes, which subsequently affect the polymer gas-transport properties. The bridging group (−O− and −C=O) between the two phenyl rings present in dianhydrides such as 3,3',4,4'-Oxydiphthalic (ODPA) and 3,3',4,4'-benzophenone tetracarboxylic (BTDA) facilitates the bond rotation, reducing the glass transition temperature Tg (Kothawade *et al.*, 2008). PIs with higher concentrations of carbonyl groups also tend to have higher CO_2 solubilities, which indicate the presence of interaction between CO_2 and carbonyl groups and increase the permeability coefficients; a larger degree of intermolecular interaction for BTDA permits more efficient polymer chain packing in the forming membranes (Tanaka *et al.*, 1992; Wang *et al.*, 2005). The mobility of the intrasegmental polymer chains can be affected by the conformation of disulfonyl anhydride segments like in the 4,4',5,5'-sulfonyldiphthalic anhydride (DS), which has low torsional mobility. Polymers containing many 6F linkages like those contained in the 4,4'-(hexafluoroisopropylidene) diphthalic anhydride (6F), have restricted intrasegmental mobility of polymer chains, which results in increased chain stiffness and typically exhibit gas permeabilities that are higher than those displayed by other polymers (Sanders *et al.*, 2012).

The properties of the segmental molecules in the PIs mentioned above encouraged us to synthesize four novel PIs from 4-fluoro-4'4"-diaminotriphenylmethane (TMF) and different aromatic anhydrides; the trade-off between the permeability and selectivity properties of these materials showed a good balance (Guzmán-Lucero *et al.*, 2015). These fluorinated polyimides showed d-spacings that were higher than those featured by other PIs reported elsewhere; the introduction of the 4-fluoro-phenyl pendant group into the polymer backbone induced significant permeability improvements. The permeability coefficients of 6F-TMF are greater than those featured by other PIs by one order of magnitude, among the synthesized PIs. The market for gas separation is moving on exploring solutions for particular applications, where modularity, plant size and process cost become important factors to design effective and efficient membrane separation systems (Brunetti *et al.*, 2015). In order to develop an industrial plant based on a new polymer for gas separation, the membranes must show operational or economic advantages, so it is important to study the effect of the transport properties of the new membranes on the design and economy of a membrane system. We have previously studied the separation properties of a PI membrane for a ternary mixture ($CH_4/CO_2/H_2S$), determining the composition, pressure and feed flow effects on the economy of the process (Palomeque-Santiago *et al.*, 2016); we have also studied the effect of the transport properties on the design of a plant and the economy of a process for a single permeator (Palomeque-Santiago *et al.*, 2017). The influence of the permeability and selectivity of different membranes on the design of an industrial plant is an important issue. The aim of this work was to study the influence exerted by the membrane properties of this new series of PIs on the gas processing costs and the size of membrane separation systems. The CH_4/CO_2 mixture was used with different CO_2 compositions in the feed; the effect of the flow rates and pressure was also studied at constant CO_2 concentration of 10 mol%.

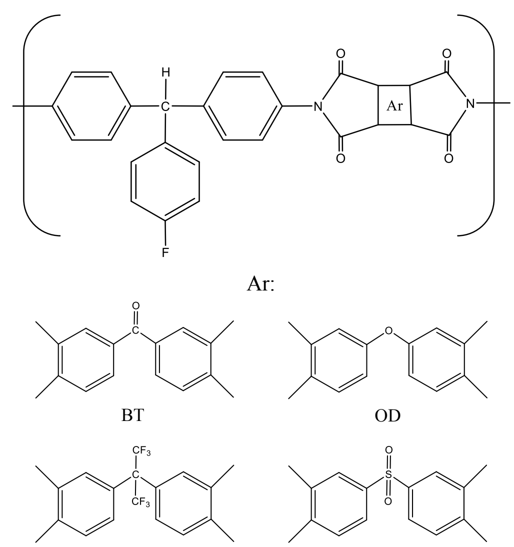

Fig. 1. Chemical structure of the polyimides (PIs).

2 Materials and methods

2.1 *Synthesis of membranes*

PIs based on 4-fluoro-4’4"-diaminotriphenylmethane (TMF) were synthesized by the high-temperature, one-step method; four dianhydrides were used: 3,3’,4,4’-oxydiphthalic dianhydride (OD), 3,3’,4,4’-benzophenone tetracarboxylic dianhydride (BT), 4,4’,5,5’-sulfonyldiphthalic anhydride (DS), and 4,4’-(hexafluoroisopropylidene)diphthalic anhydride (6F), all from Sigma-Aldrich. Dense membranes of aromatic PIs from these dianhydrides and TMF were prepared as previously reported (Guzmán-Lucero *et al*., 2015); the chemical structure of the synthesized PIs are shown in Fig. 1. The gas transport studies were conducted using a standard constant-volume, variable-pressure apparatus. Permeability data were determined from the slope of the downstream pressure vs. time plot after steady state had been achieved; Table 1 reports the transport properties of these membranes.

2.2 *Simulation study*

The gas transport properties of the TMF PIs obtained in Section 2 were subjected to simulation studies in order to establish the influence of these properties on the design and economy of an industrial membrane system for gas sweetening; Matrimid® PI was included for comparison purposes. This study was performed with the commercial software ProII 9.0, which simulates the fractionation of components across a semi-permeable membrane. The model incorporated by ProII 9.0 is applicable to high flux asymmetric membranes with any flow pattern, provided the assumptions described by Pan (1983). The membrane separator simulates the fractionation of component vapor across an asymmetric semi-permeable membrane consisting of a thin skin and a porous substrate. The permeating components pass through the skin and flow through the porous substrate to form a bulk permeate product; this permeate stream contains most of the CO_2 separated from the feed, generating a disposable stream. The components that do not cross the membrane form the retentate (product) on the feed side of the membrane.

2.2.1 *Model description*

The controlling equation for the model is:

$$F_i = K_i \times Area \times (P_{i\ retentate} - P_{i\ permeate}) \quad (1)$$

where:
F_i = Permeation flow of component i through the membrane (std volume / time)
K_i = Permeability constant of component i (volume*thickness / [area*time*pressure])
$Area$ = Effective permeable area of the membrane
P_i = Partial pressure of component i

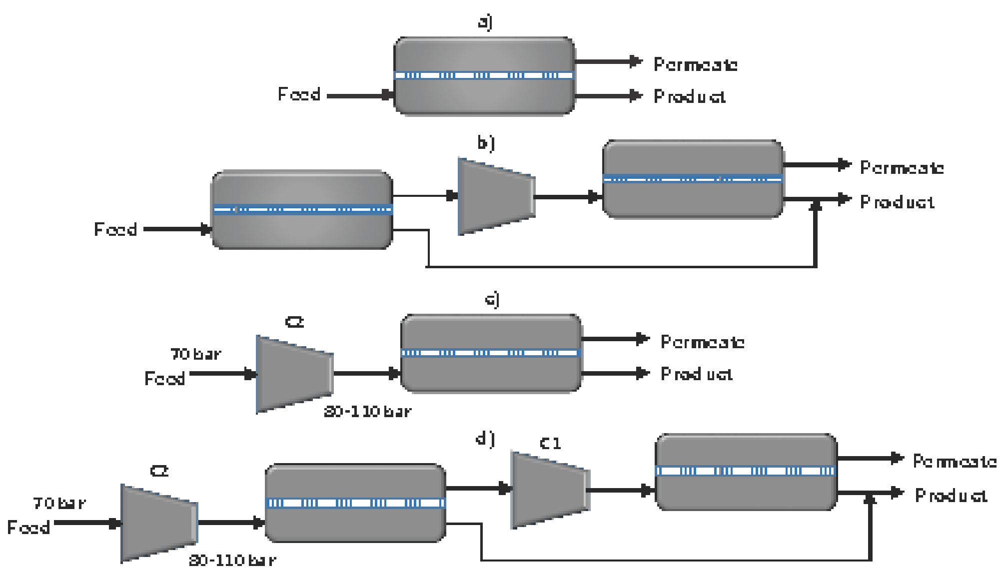

Fig. 2. Flow schemes: a) single stage, b) double stage, c) single stage with a feed compressor, d) double stage with a feed compressor.

Table 1. Gas transport properties of polyimides and total membrane module cost.

Polyimide	Permeability*		Ideal selectivity*	Module Cost
	CO_2 (10^{+7})	CH_4 (10^{+9})	CO_2/CH_4	$\$/m^2$ ($\$/ft^2$)
BT-TMF	2.0	7.6	26.7	75 (7)
OD-TMF	1.6	5.7	27.8	86 (8)
DS-TMF	2.2	6.9	31.0	86 (8)
6F-TMF	22.2	54.0	41.2	118 (11)
Matrimid®**	6.3	17.8	35.7	75 (7)

*Permeability in $m^3 \cdot m/(m^2 \cdot day \cdot bar)$.

** Sanders *et al*., 2013.

2.2.2 *Assumptions*

The following assumptions are provided:

1. The feed gas is on the skin side of the asymmetric membrane and is well-mixed.
2. There is constant pressure on both sides of the membrane. Feed gas pressure drop is negligible.
3. No mixing of permeate fluxes of different compositions occurs inside the porous supporting layer of the membrane.
4. The porous supporting layer has negligible resistance to gas flow, and diffusion along the pore path is insignificant due to high permeate flux.
5. The permeabilities of gases through the membrane are independent of pressure, concentration and mixed-gas effects. There are no effects by CO_2 plasticization.
6. Pretreatment of the feed stream is not considered.

2.2.3 *Configurations*

Single and double stage process configurations were considered. The flow diagrams are illustrated in Fig. 2. Single stage is the first option to take into consideration, since it is the simplest and the cheapest; when the single stage is not satisfactory for some particular needs, another permeator can be added, where the low-pressure stream is recompressed and fed to a second permeator connected in series; the permeates of the first and the second stages are mixed to get the final product. The area of the second stage was calculated to get a product into specification of 2 mol% CO_2 (Fig. 2b). The considered natural gas composition was a binary CH_4/CO_2 gas mixture containing 5, 10, 20, 30 and 40 mol% CO_2, which were set as typically used in gas processing plants.

2.2.4 *Operating conditions*

Processing capacity: 40 MMSCFD
Feed pressure: 70 bar
Permeate pressure: 3 bar
Feed temperature: 25 °C

The target was set to obtain a CO_2 composition of 2 mol% in the product stream to meet pipeline specifications. The input variables introduced to the simulator are the operating conditions and gas transport properties of Table 1. The areas of the permeators are variables to be calculated. The simulator computes, among other properties, the composition and the flow rates of all streams, as well as the compressor work under the specified conditions.

2.2.5 *Economic parameters*

Several authors have published economic parameters to make evaluations of membrane processes, and differences among them can be found (Hao *et al*., 2002; Qiu *et al*., 1989; Bhide and Stern, 1993; Data and Sen, 2006), but these methodologies provide the bases for estimating particular cases by updating costs, market conditions, prices of hydrocarbons, and local factors. Cost estimations in this study were performed by following the methodology by Hao *et al*., (2008) who assumed that gas processing costs are mainly determined from the total plant investment (TPI), annual variable operating and maintenance costs (VOM) and annual cost of CH_4 lost in the permeate (CH_4LS). In this study, hollow fiber modules were considered, and for determining the membrane module cost, an estimation of the production cost of the synthesized PIs was performed according to the price of the raw materials; the costs of our polymers are in the range of high performance materials: 1-10 USD/m^2 (0.09-0.93 USD/ft^2) (Baker and Lokhandwala, 2008). The estimated membrane module costs are shown in Table 1.

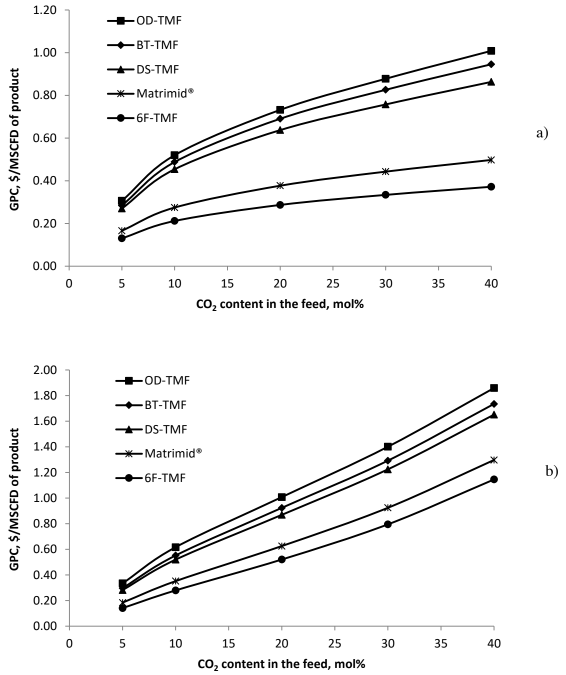

Fig. 3. Gas processing costs vs. CO_2 content in the feed for: a) single stage, b) double stage.

3 Results and discussion

3.1 *Effect of feed composition on GPC*

Our results may have a deviation of about 15% because of the assumptions listed in the section 3.2. As shown in Fig. 3, gas processing costs increase with the CO_2 content in the feed, according to other authors (Bhide and Stern, 1993; Hao *et al*., 2008). This is because the total plant investment (TPI), annual variable operating and maintenance cost (VOM) and annual cost of CH_4 lost in the permeate (CH_4LS) also increase as a result of the higher membrane area required for achieving more CO_2 removal; it is worth noting that lower gas processing costs (GPC) are obtained with a single stage. The addition of a second permeator is advisable for recovering more methane, thus lowering its annual cost lost in the permeate, however higher GPC values obtained with a double stage are due to the increase in the operating and maintenance costs and higher plant investment. These factors are related to the addition of the second compressor and an extra-module; the savings obtained by the reduction of methane loss in the permeate cannot fully compensate these costs. The membrane with the smallest GPC was the 6F-TMF related to the smallest area to reach the goal of 2 mol% CO_2 in the product stream; this material also had the highest values of methane recovery, followed by the Matrimid® membrane.

3.2 *Effect of feed flow on GPC*

The size of the membrane processes is a key factor when this technology is proposed for gas separations so it is important to determine how the GPC is going to change when deciding an increase in the plant capacity. In some applications such as CO_2 capture or biogas purification, small membrane plants are appropriate (Qiu *et al*., 1989; Brunetti *et al*., 2014), but for natural gas processing, the size of the industrial plant is very important, since high flows are obtained either from the gas wells or as associated gas. Gas processing complexes spread and gather the available streams, which are prepared for the design conditions. One of the advantages of the membrane processes is that they feature a modular design which permits easy expansion or operation at partial capacity; a small plant can be expanded to higher capacities by installing more modules.

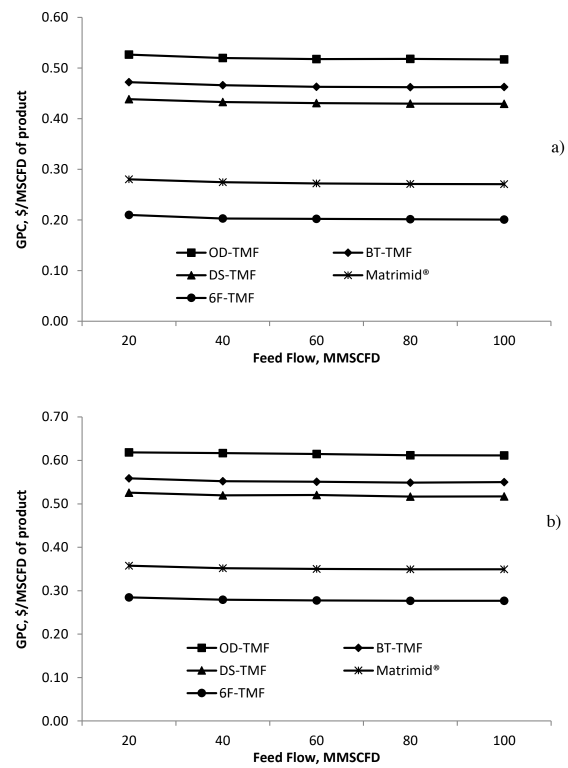

Fig. 4. Gas processing costs as a functions of feed flow for: a) single stage, b) double stage.

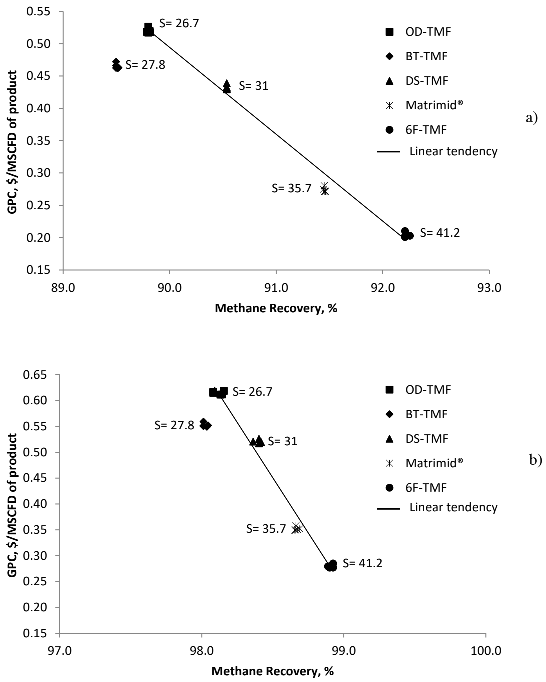

Fig. 5. Relationship between gas processing costs and methane recovery as a functions of the feed flow for: a) single stage, b) double stage.

The feed flow effect for the five membranes was simulated from 20-100 MMSCFD, maintaining 70 bar in feed pressure and a 90/10 CH_4/CO_2 composition. As seen in Fig. 4, the GPC is constant with the increase in feed flow with higher values. Lababidi *et al*. found the lowest operating cost at the highest feed flow rate due to the savings on the largest total amounts of enriched methane; then, a higher methane recovery was obtained (Lababidi *et al*., 1996). In our case, methane recoveries were constant with the feed flow because the target was set at 2 mol% CO_2 in the product stream (retentate), so the methane recovery was also constant. Bhide and Stern reported that for a three-stage membrane process with retentate recycle; the separation costs for the membrane process decreased with an increase in the feed flow rate at any given CO_2 concentration in the feed, but these differences were very small at a constant mol fraction of CO_2 in the feed (Bhide and Stern, 1993). Subsequently, Hao *et al*. found that for a configuration without recycle, as in our case, gas processing costs are constant when the CO_2 concentration in the feed increases above a certain point depending on the feed flow rate (Hao *et al*., 2008). An increase in the plant capacity implies investments in equipment (membrane area and compressor), but these facts have no influence on GPC. Since methane recovery is constant with the feed flow, this behavior resulted in dots for each membrane in a GPC vs. methane recovery diagram; each dot represents a behavior pattern at each feed flow (20-100 MMSCFD). A straight line was obtained as a function of membrane selectivity (Fig. 5).

3.3 Effect of feed pressure on GPC

When a new plant is to be designed, the stream to be treated could be considered as a mixture of several streams, coming from several extraction wells, under specific conditions. Then, it is conditioned to a feed pressure and its composition is almost constant with time and normally for several years. In membrane processes, the increase in feed pressure leads to a decrease in the required membrane area (Palomeque-Santiago *et al*., 2016; Hussain and Hägg, 1996); then, savings in permeator equipment are obtained; on the other hand, lower methane loss in the permeate and higher methane recovery are obtained. The effect of pressure on gas processing costs has been studied by several authors (Bhide and Stern, 1993; Hao *et al*., 2008; Safari *et al*., 2009).

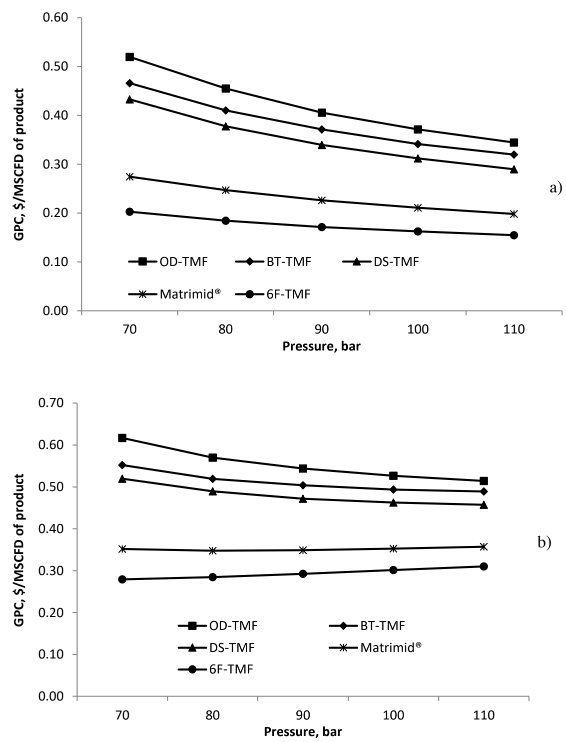

Fig. 6. Gas processing costs as a functions of pressure for: a) single stage, b) double stage.

Table 2. Results obtained as a function of the feed pressure at 40 MMSCFD, 10 mol% CO_2.

	Single Stage		Double Stage		
Feed Pressure (bar)	Total Membrane Area(m^2)	Compressor work 2 (Kw D)	Total Membrane Area (m^2)	Compressor work 1 (Kw D)	Compressor work 2 (Kw D)
OD-TMF					
70	84,300	0	102,300	1,126	0
80	69,400	169	83,400	1,197	169
90	58,400	324	70,400	1,259	324
100	50,600	467	60,700	1,324	467
110	44,500	600	53,000	1,384	600
BT-TMF					
70	65,200	0	79,000	1,145	0
80	53,500	169	64,500	1,213	169
90	45,300	324	54,700	1,281	324
100	39,100	467	47,200	1,343	467
110	34,500	600	41,500	1,407	600
DS-TMF					
70	64,000	0	76,300	1,083	0
80	52,200	169	62,300	1,146	169
90	44,000	324	52,400	1,208	324
100	38,000	467	45,200	1,270	467
110	33,300	600	39,600	1,326	600
6F-TMF					
70	6,760	0	7,890	980	0
80	5,450	169	6,350	1,035	169
90	4,550	324	5,300	1,090	324
100	3,920	467	4,540	1,148	467
110	3,410	600	3,950	1,197	600
Matrimid®					
70	3,090	0	26,730	1,028	0
80	18,500	169	21,700	1,091	169
90	15,500	324	18,200	1,148	324
100	13,300	467	15,600	1,204	467
110	11,600	600	13,600	1,256	600

Since the membrane area is inversely proportional to the pressure difference on both sides of the membrane, according to Eq. (1), when feed pressure increases, the required membrane area lowers, impacting on all economic parameters, especially on total plant investment and on operational and maintenance costs, and as a result, GPC lowers.

The results for the five membranes are illustrated in Fig. 6 for the configurations of Figs. 2a and 2b. Higher GPC values are obtained with a double configuration because of plant investment in membrane areas and compression equipment. The membranes with lower selectivity (OD-TMF, BT-TMF and DS-TMF) gave higher GPC values than the membranes with higher selectivities (Matrimid® and 6F-TMF). For the first group, the GPC tendency was to decrease with pressure as it has been well established; for the second group, the membranes with higher selectivity presented the same tendency to decrease for a single configuration, but in the double configuration, the GPC data for the Matrimid® membrane became constant and for 6F-TMF, GPC has a slight tendency to increase. Most of the analyses from the literature assume that the feed pressure is a default variable, however, for a specific stream under specific conditions, if lower membrane area is pursued by increasing the feed pressure, the pressure increase must be gotten by an additional compressor, so the savings in membrane area are canceled to a certain extent by additional compressor costs. In this study, it was assumed that a specific stream was available at 70 bar with a CO_2 concentration of 10 mol%, and it was desirable to design a plant to process 40 MMSCFD without sacrificing gas processing costs. The effect of feed pressure was studied by increasing the pressure from 70 to 80, 90, 100 and 110 by adding a second compressor to the feed stream. Gas processing costs were calculated by taking into account this second compressor (Figs. 2c and 2d). The results showed a clear tendency to increase GPC as pressure increases for all the membranes for a double configuration, and for a single permeator this tendency is only observed in the Matrimid® and 6F-TMF membranes, a behavior pattern that has not been reported before (Fig. 7).

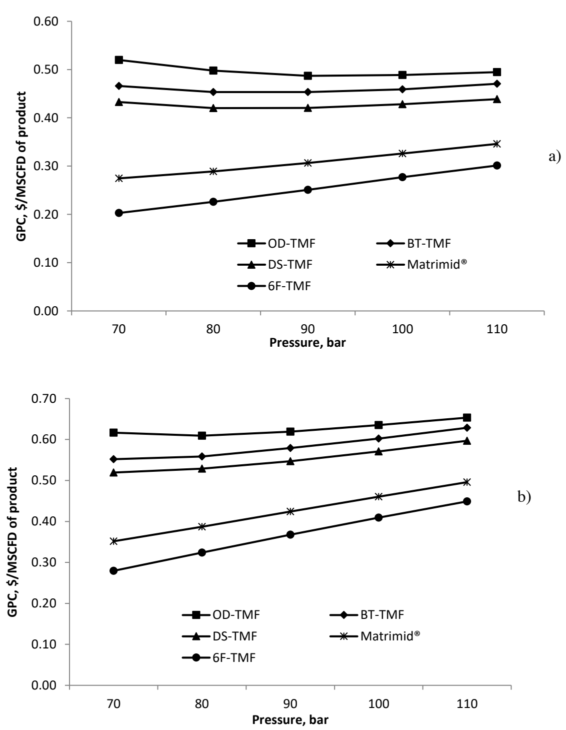

Fig. 7. Gas processing costs as a functions of pressure with an additional compressor at the feed stream for: a) single stage, b) double stage.

Table 3. Economic data as functions of the feed pressure.

Pressure (bar)	TPI (MM$)	VOM (MM$/year)	GPC ($/MSCF)
OD-TMF			
70	22.89	2.67	0.617
80	21.82	2.40	0.570
90	21.16	2.22	0.544
100	20.88	2.10	0.527
110	20.76	2.01	0.514
6F-TMF			
70	11.64	0.93	0.279
80	12.09	0.94	0.285
90	12.59	0.96	0.293
100	13.15	0.99	0.302
110	13.64	1.02	0.310

This opposite trend can be explained as follows: the increase in pressure exerts an important effect on the membranes with low CO_2/CH_4 selectivity, for example, the membrane area is reduced in 39,800 m^2 from 70 to 110 bar for the OD-BT membrane and this reduction is of only 3,350 m^2 for the 6F-TMF membrane for the same pressure difference (single stage, Table 2), so the savings in membrane area exerts a slight effect on this last membrane; in addition, the compression work is in the same order for both membranes, giving as a result that the TPI and VOM decrease for the first membrane and they increase for the second one as illustrated in Table 3. On the other hand, the investment in the second compressor is higher than the savings in membrane area due to the pressure increase, which is an important factor for making decisions in the design of plants.

In this study, it was considered that the permeators were considered to be constructed with hollow fibers, which are cheaper than spiral wounds (Baker, 2004). A calculation was performed increasing the membrane module costs for the case of spiral wounds around 4.5 times of the cost of hollow fibers. The gas processing costs increased from 21 (6F-TMF) to 58% (OD-TMF) for a single stage and from 17 (6F-TMF) to 56% (OD-TMF) for a double stage. In these cases, the total plant investments due to membrane area were higher than the investments in compressor. The GPC showed a marked tendency to decrease as the pressure increased for the double configuration, which is an opposite trend to that obtained with a lower membrane module cost for hollow fibers in this study and the normal tendency reported by other authors.

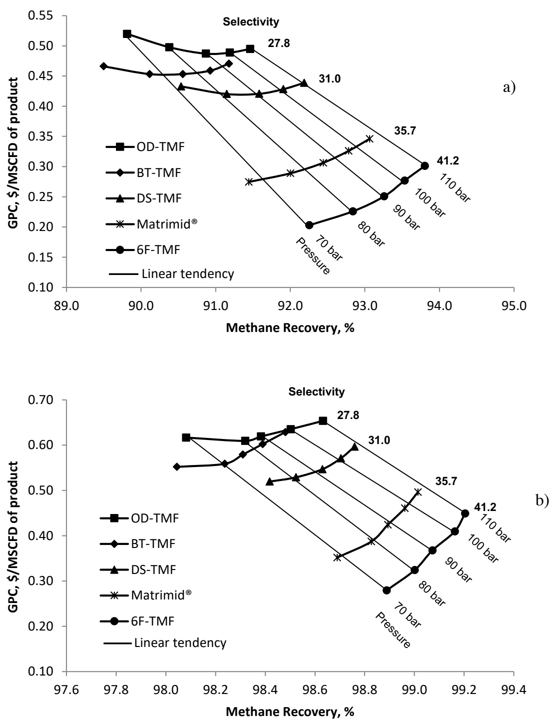

Fig. 8. Relationship between gas processing costs and methane recovery as a functions of pressure for: a) single stage, b) double stage.

These results prove the great influence of adding a second compressor to the feed stream.

There are two important issues that refiners pay attention to in separation processes with membranes: the cost of separation and recovery of the main product to be commercialized. The relationship between methane recovery and gas processing cost is illustrated in Fig. 8 as a function of pressure. A linear tendency is observed at constant pressure and a function of membrane selectivity; the BT-TMF membrane shows a different behavior. A more selective membrane permeates more CO_2 and the purity of the product increases, so higher methane recovery is obtained, which is reflected in lower GPC values.

Conclusions

The membrane properties of a new family of PIs based on 4-fluoro-4'4"-diaminotriphenylmethane and different dianhydrides showed a good balance between permeability and CO_2/CH_4 selectivity. These transport properties exerted an important effect on the economy of membrane plants for gas sweetening. The membrane areas needed to get a product into pipeline specification (2 mol% CO_2) from different feed compositions are closely related to the permeabilities of the membranes. 6F-TMF, a fluorinated membrane in the dianhydride and diamine moieties, exhibited the best performance with lower membrane area requirements, higher methane recoveries, lower methane loss in the permeate, and lower gas processing costs (GPC). The cost of manufacturing this membrane is the highest among the materials under study, but even so, the savings in the process compensate this cost. At constant pressure and variable feed flow, GPC follows a linear tendency with the membrane selectivity; the same linear tendency is obtained with variable pressure. GPC remained constant at any feed flow value; this was due to the constant methane recovery, fixed by the constant target in the product stream, 2 mol% CO_2. The normal behavior is that GPC decreases with the increasing feed flow due to the savings in methane recovery, but in our case these savings remained constant.

The influence of pressure on the design parameters was elicited by the addition of a second compressor to the feed stream in order to increase it from 70 to 80, 90, 100 and 110 bar since pressure was not considered as a default variable; it was assumed that the design was based on a real feed stream conditioned to be treated in a gas processing complex. The additional expenses regarding the investment and operational and maintenance costs of the compressor, annulled the positive effect of pressure on the membrane area decrease, causing the gas processing cost to increase with pressure in the double configuration. This effect was more evident with the 6F-TMF membrane, which had the highest selectivity among the studied membranes. Another feature that influences this opposite behavior was the low cost the membrane module made of hollow fibers; an analysis with more expensive spiral wound permeators was carried out, where the investment in membrane area was higher than the investments in compressor; then, the GPC decreased with the increase in feed pressure as reported in the literature.

Acknowledgements

The authors acknowledge the Instituto Mexicano del Petróleo for supporting this research work (Project D.0264).

Abbreviations

MSCFD	103 standard cubic feet per day (at 14.696 psia and 60 °F)
MMSCFD	106 standard cubic feet per day (at 14.696 psia and 60 °F)

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