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Maryam Takht Ravanchi[a][b]; Tahereh Kaghazchi[a]; Ali Kargari[c]
[a] Department of Chemical Engineering, Amirkabir University of Technology (Tehran Polytechnic), Tehran, Iran [b] National Petrochemical Company, Research and Technology Co., Tehran, Iran [c] Department of Mechanical Engineering, K. N. Toosi University of Technology, Tehran, Iran

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Separation of Propylene-Propane Mixture Using Immobilized Liquid Membrane via Facilitated Transport Mechanism

Maryam Takht Ravanchi,[1,2] Tahereh Kaghazchi,[1] and Ali Kargari[3]
[1]Department of Chemical Engineering, Amirkabir University of Technology (Tehran Polytechnic), Tehran, Iran
[2]National Petrochemical Company, Research and Technology Co., Tehran, Iran
[3]Department of Mechanical Engineering, K. N. Toosi University of Technology, Tehran, Iran

Abstract: Separation of propylene-propane mixtures using immobilized liquid membrane was investigated. A porous polymeric sheet was used as support to immobilize the liquid membrane. The effect of propylene partial pressure in feed stream, trans-membrane pressure, and carrier concentration on membrane separation performance was investigated and the results were evaluated in terms of separation factor, propylene permeability, and propane permeability. Propylene permeability ranged from 0.4 to 650 Barrer. Moreover, it was observed that for 30:70 (vol.%) propylene-propane mixture, at pressure 120 kPa and carrier concentration 20 wt.%, a separation factor of 480 was obtained.

Keywords: Facilitated transport, permeability, propylene-propane separation, silver nitrate, supported liquid membrane

INTRODUCTION

Light olefins such as ethylene and propylene are produced in great quantities in petrochemical plants. An important step in the manufacture

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Address correspondence to Tahereh Kaghazchi, Department of Chemical Engineering, Amirkabir University of Technology (Tehran Polytechnic), No. 424, Hafez Ave., PO Box 15875-4413, Tehran, Iran. Tel.: +98-21-64543152; Fax: +98-21-66405847. E-mail: Kaghazch@aut.ac.ir

of olefins is large-scale separation of the olefin from the corresponding paraffin. Currently, this separation is carried out by distillation, which is highly energy-intensive due to the cryogenic temperatures required for the process. Distillation columns are often up to 100 meter tall and typically contain over 200 trays. With reflux ratios greater than 10, a very high energy input is required for the distillation process (1,2).

Nowadays, membrane technologies are becoming more frequently used for the separation of wide varying mixtures and can compete with traditional schemes (3–13).

Membrane technology has been proposed as an alternative approach to the conventional distillation process for olefin/paraffin separation (14–17). Polymeric membranes have been developed for olefin/paraffin separation. However, their performance for propylene/propane separation is particularly poor. Currently, the best polymeric membranes exhibit propylene/propane separation factors of less than 10, which is still insufficient for practical use (Table 1).

A very effective way to increase the membrane selectivity is to incorporate some carrier, which reacts with a specific permeate reversibly and selectively, in the membrane. This type of membrane is called facilitated transport membrane (FTM). The most commonly used facilitated transport membrane types are immobilized liquid membranes (ILMs) and solvent-swollen fixed-site carrier membranes. ILMs, which are also named supported liquid membranes (SLMs), are made by impregnating a microporous membrane with a solution containing the carrier. The carrier solution is held within the pores of the membrane by capillary forces

Table 1. C_3H_6/C_3H_8 permeation data in polymeric membranes

Polymer[a]	Feed pressure	Separation factor	Reference
Matrimid-Thermid 85/15	2–3 bar	4	18
PPO	2–4 bar	4.25	19
EC	3–3.9 atm	3.25[b]	20
CA	3–3.9 atm	2.6[b]	20
PSF	3–3.9 atm	1.4[b]	20
6FDA-TeMPD	2 atm	8.6	21
PPO	2 atm	9.1	21
P4MP	2 atm	2	21
1.2PB	2 atm	1.7	21
PDMS	2 atm	1.1	21

[a] A detailed chemical description of the abbreviations used for polymer materials is given in (1).

[b] Feed mixture: 55 vol.% propylene, 45 vol.% propane.

(22,23). Nonporous solvent-swollen fixed-site carrier membranes have been used to improve the mechanical stability of facilitated transport membranes. Water-swollen fixed-site carrier membranes exhibit excellent separation properties. However, unless water and/or plasticizers, such as glycerin, are present in the polymeric membrane matrix, tight ion-paring of the silver salt occurs, resulting in very low mobility of the carrier species and, consequently, very low gas fluxes. As a result, fixed-site carrier membranes must be operated continuously in a water-vapor-saturated environment. The addition of water vapor to the feed and permeate stream and the required subsequent removal of water vapor from the olefin-rich permeate stream make the use of these membranes impractical for industrial applications. Solid polymer electrolytes are an alternative type of facilitated transport membrane (24). In these systems, the salt dissolves in a polymer matrix in the solid state and dissociates into anions and cations. The essential feature that distinguishes a solid polymer electrolyte from polymer/salt systems based on fixed-site carrier membranes is that ionic motions in a solid polymer electrolyte take place without a solvent or plasticizer being present (25). Some of the results of propylene-propane separation using solid polymer electrolyte membranes are summarized in Table 2.

In order to improve the performance of solid polymer electrolyte membranes, some researchers used a third component such as amino acid, surfactant, or phthalates in the membrane system. The implication of such additive in the membrane will improve the long term stability of the electrolyte membrane. An overview of these membrane systems can be found in literature (31–36).

In this paper, the performance of an immobilized liquid membrane system for the separation of propylene-propane mixture was investigated.

FACILITATED TRANSPORT IN MEMBRANE

It is well known that some transition metals react reversibly with alkenes in the solution. The ability of the transition metal ion as a carrier is largely dependent on the intensity of the π-complexation with alkenes, which in turn is determined primarily by the electronegativity. If the electronegativity of the metal is excessively high, the metal is not suitable for the facilitated carrier because it is susceptible to irreversible reaction with the π electrons of the alkene; otherwise, if the electronegativity is too low, the metal is impractical as a carrier due to its weak interaction with the alkene. For reversible reaction between the transition metal ion and the alkene, the electronegativity of the metal is preferably in the range of 1.6 to 2.3. In the facilitated transport membrane, the transition metal

Table 2. C_3H_6/C_3H_8 permeation data in solid polymer electrolyte membranes

Membrane[a]	Mole fraction of Ag	Separation factor	Reference
PMMA/$AgBF_4$	0.5	40[b]	26
PMMA/$AgCF_3SO_3$	0.5	18[b]	26
PBMA/$AgBF_4$	0.5	35[b]	26
PBMA/$AgCF_3SO_3$	0.5	14[b]	26
PVMK/$AgBF_4$	0.5	54[b]	26,27
PVMK/$AgCF_3SO_3$	0.5	15[b]	26
PVP/$AgBF_4$	0.5	50[b]	26
PVP/$AgCF_3SO_3$	0.5	15[b]	26
POZ/$AgBF_4$	0.5	48[b]	26
POZ/$AgCF_3SO_3$	0.5	17[b]	26
PEOx/$AgBF_4$	1	58[b]	28
PEOx/$AgCF_3SO_3$	1	17[b]	28
PVP/$AgBF_4$	1	68[b]	28
PVP/$AgCF_3SO_3$	1	15[b]	28
PVA/$AgSbF_6$	0.2	130	29
PAAm/$AgBF_4$	0.67	170[b]	30

[a] A detailed chemical description of the abbreviations used for polymer materials is given in (1).

[b] Feed mixture: 50 vol.% propylene, 50 vol.% propane.

salt with lattice energy of less than 2500 kJ/mol is preferable in order to reduce the tendency of the anion of the transition metal salt to form a strong ion pair with the cation. Based upon these criteria, silver (Ag^+) salt is selected as carrier in facilitated transport membrane (37).

Facilitated transport membranes differ from conventional polymer membranes by employing a carrier that selectively forms a complex with one of the components in the feed gas. Gas permeation across a facilitated transport membrane takes place by two mechanisms: first, solution/diffusion of free gas molecules, and second, diffusion of the carrier-gas complex. The second mechanism occurs only for gases that can react chemically with the carrier. The total flux of the membrane is the sum of the flux of the carrier-gas complex and the flux of the free gas molecules (38).

The mechanism by which facilitated transport takes place in a liquid membrane containing silver ions as carrier is illustrated schematically in Fig. 1 for the separation of olefin/paraffin mixtures. On the high-pressure side of the membrane, both olefin and paraffin are absorbed into the membrane. However, only olefin forms a complex with the silver-ion carrier. Desorption of olefin at the low-pressure side of the membrane leads to dissociation of the olefin–silver complex. The olefin flux of the membrane

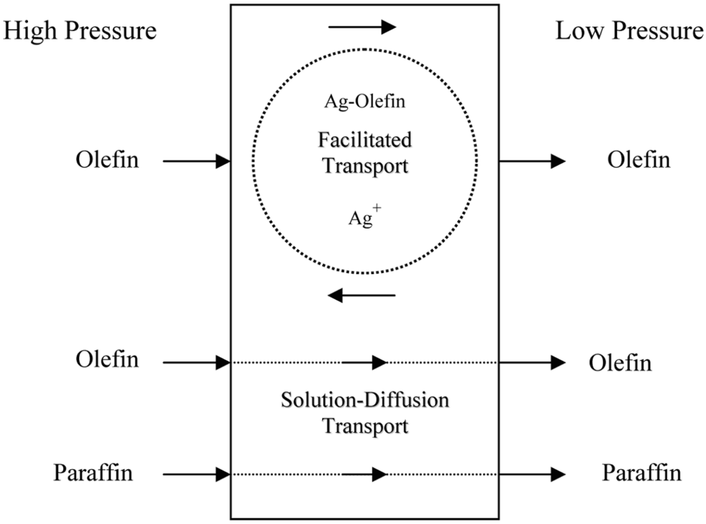

Figure 1. Schematic representation of a facilitated transport mechanism.

depends on the concentration of the silver ions and the equilibrium constant for the reaction between the silver ion and olefin. Figure 1 implies that the olefin–silver complex is mobile and diffuses across the membrane acting as a shuttle for the transport of olefin (39,40).

EXPERIMENTS

Feed Gases

Industrial grade propylene from Tabriz Petrochemical Company and industrial grade propane from Tehran Refinery Complex were used as feed gases and pure nitrogen was used as sweep gas. The detailed analysis of the feed gases is presented in Table 3.

Carrier Solution

Silver Nitrate ($AgNO_3$, GR Pro Analysis) which was purchased from Merck Co. was used as the carrier of propylene. An aqueous solution of silver nitrate was prepared by dissolving silver nitrate in deionized water.

Table 3. The detailed analysis of feed gases

Component	Propylene	Propane
N_2	–	–
CH_4	<1 ppm	5 ppm
C_2H_6	<1 ppm	545 ppm
C_2H_4	<1 ppm	1 ppm
C_3H_8	2622 ppm	99.79 mole%
C_3H_6	99.74 mole%	731 ppm

Flat Sheet Membrane Module

Schematic diagram of the membrane module is shown in Fig. 2. The lower and upper compartments, with inner diameter 110 mm, were made from stainless steel (AISI 316). Hydrophilic polyvinilydene diflouride (PVDF) flat sheet membranes (Durapore from Millipore, thickness 125 µm, pore size 0.22 µm, filter diameter 142 mm) were used as the support of the liquid membrane. After being immersed in the carrier solution, the membrane filter was sandwiched between two compartments of the module. Once prepared, the membrane filter could be used for 3–4 weeks with no change in separation and permeation properties.

Experimental Apparatus and Procedure

The schematic diagram of the experimental setup is shown in Fig. 3. All tubing used to connect all parts of the setup was stainless steel (AISI 316). The experimental procedure is as follows.

Propylene and propane, after passing through mass flow controllers (Brooks Instruments, model 5850S), were mixed and entered the humidifier. The humidified feed passed through a temperature control system

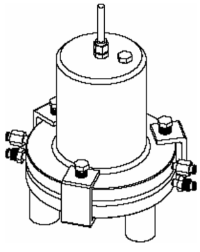

Figure 2. Schematic diagram of membrane module.

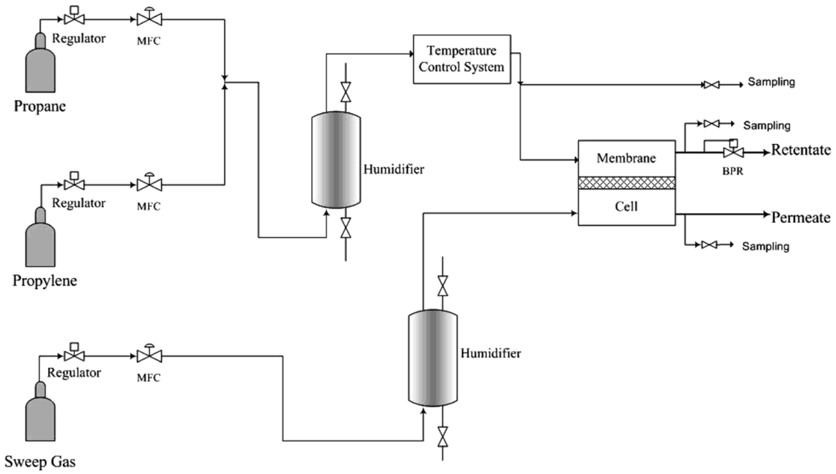

Figure 3. Schematic diagram of the experimental setup.

and entered the membrane cell. A combination of a heater and a cooler were used as the temperature control system. The feed gas was introduced to the upper compartment of the cell and the sweep gas, nitrogen, was supplied to the lower compartment. The main product, permeate, was collected from the lower compartment and the secondary product, retentate, was collected from the upper compartment. A back pressure regulator (BPR, Tescom, Germany) was used on the retentate line to control the pressure of the system. During all experiments, sweep gas was at atmospheric pressure. The experiments were conducted at room temperature (298 ± 5 K). All the experimental data were obtained after an initial permeation period of 4–6 hr.

Analysis

The gas composition was determined by a Gas Chromatograph (Agilent 6890N) equipped with a Flame Ionization Detector (FID, Agilent Technologies Inc. column, HP Al/S, 0.53 mm in diameter, and 50 m in length).

Calculation of Membrane Performance

There are two important technical terms that are used for the evaluation of membrane gas separation performance; "permeability" and "separation factor."

The permeability of species i through a microporous membrane is calculated as:

$$Q_i = \frac{N_i L}{S_t \Delta P_i}$$

where:

Q_i: permeability of component i

N_i: volumetric permeation rate of component i at standard pressure and temperature

L: membrane thickness

S_t: total exposed membrane area

ΔP_i: partial pressure difference of component i across the membrane

Separation factor is defined as:

$$\alpha_{AB} = \frac{y_A^p / y_B^p}{y_A^f / y_B^f}$$

where

y_i^p: mole fraction of component i in the permeate stream

y_i^f: mole fraction of component i in the feed stream

RESULTS AND DISCUSSION

Effect of Trans-membrane Pressure

The effect of trans-membrane pressure on membrane performance is shown in Figs. 4–12. Different mixtures of propylene-propane were used as feed gas. As it can be seen, trans-membrane pressure and separation factor are directly proportional and increasing the trans-membrane pressure will cause an increase in the separation factor and a more purified product will be obtained.

Facilitated transport is a combination of two processes: absorption (on the feed side) and stripping (on the permeate side). Increasing the pressure is in favor of absorption and decreasing the pressure is in favor of stripping. Thus, increasing the feed pressure increases the absorbed propylene on the feed side. Due to the pressure difference between the feed side and the permeate side, the complexed propylene is decomplexed on the permeate side. Therefore, the more the trans-membrane pressure, the more the driving force for separation. Based upon this fact, more propylene was transported across the membrane and separation factor and propylene permeability was increased.

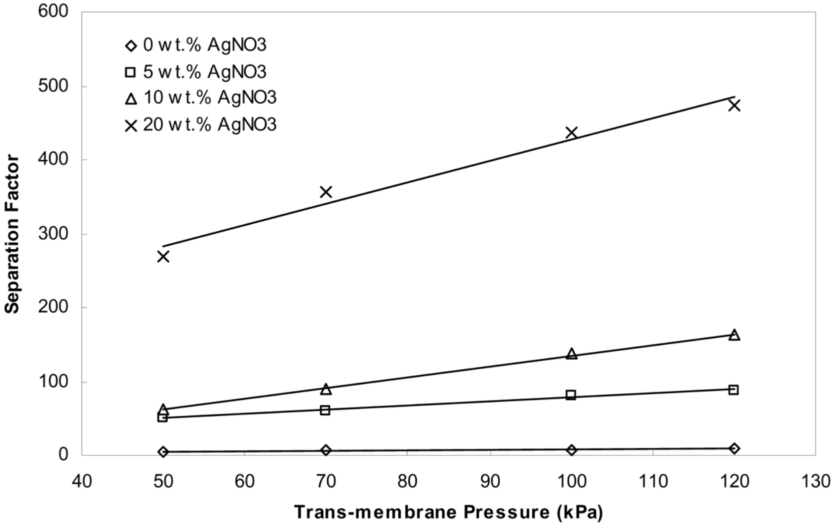

Figure 4. The effect of trans-membrane pressure on separation factor for 30:70 (vol.%) propylene-propane mixture and at different carrier concentrations.

During all of the separation experiments, pressure at the permeate side kept at a constant value.

Propane permeated via Fickian diffusion. The more the trans-membrane pressure, the more the driving force for propane permeation.

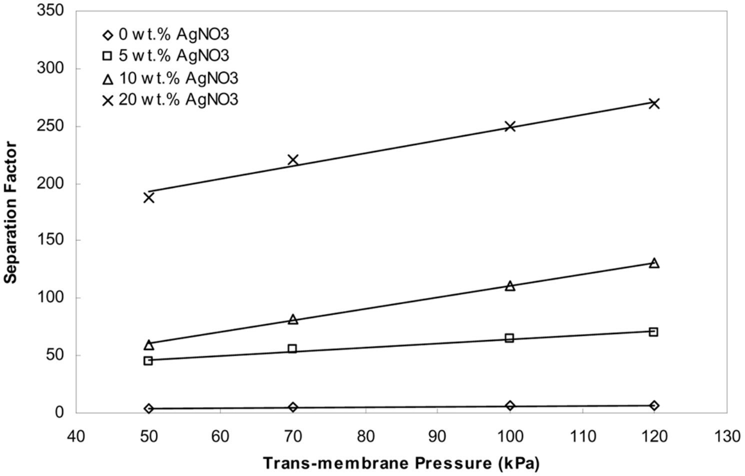

Figure 5. The effect of trans-membrane pressure on separation factor for 50:50 (vol.%) propylene-propane mixture and at different carrier concentrations.

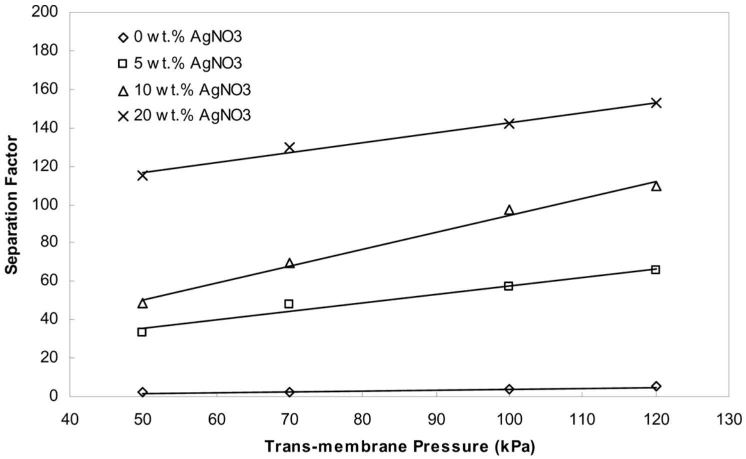

Figure 6. The effect of trans-membrane pressure on separation factor for 70:30 (vol.%) propylene-propane mixture and at different carrier concentrations.

The results of Figs. 10–12 are in agreement with this fact. For a specified feed mixture and at constant carrier concentration, increasing the trans-membrane pressure increases the propane permeability.

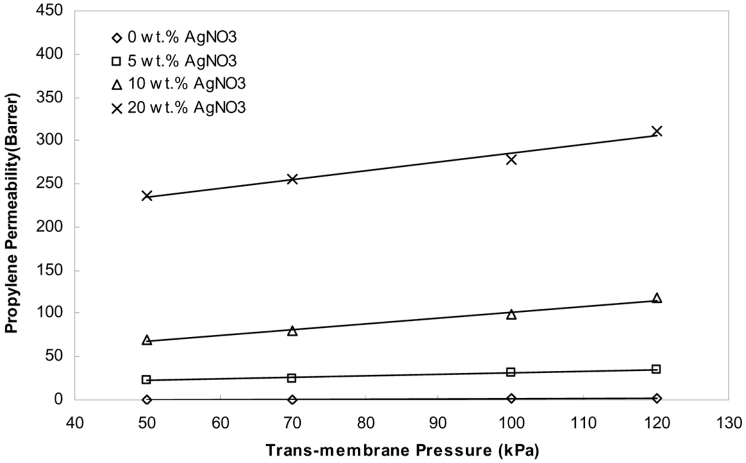

Figure 7. The effect of trans-membrane pressure on propylene permeability for 30:70 (vol.%) propylene-propane mixture and at different carrier concentrations.

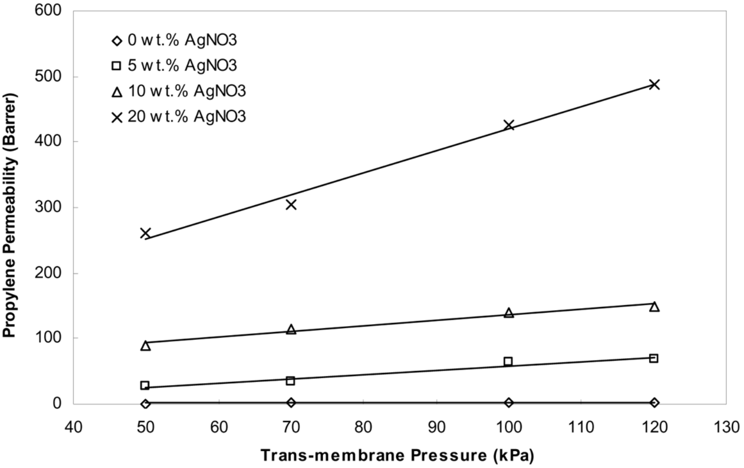

Figure 8. The effect of trans-membrane pressure on propylene permeability for 50:50 (vol.%) propylene-propane mixture and at different carrier concentrations.

Effect of Carrier Concentration

The effect of carrier concentration on membrane performance is shown in Figs. 13–15. As it is obvious, carrier concentration and separation factor are directly proportional confirming that olefin transport results from interaction of the olefin with carrier ions.

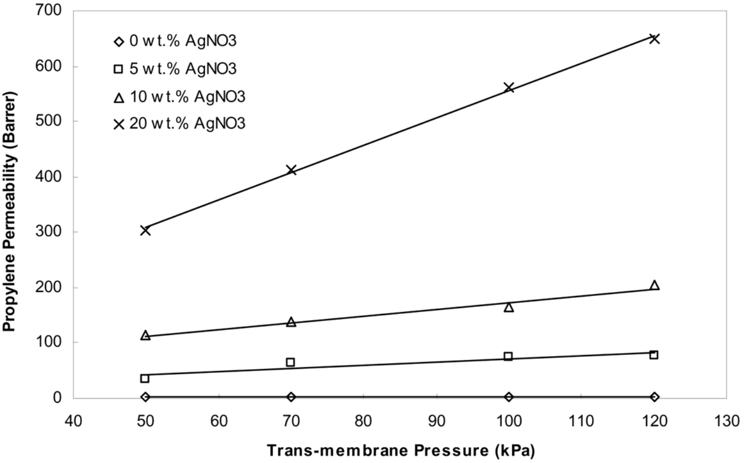

Figure 9. The effect of trans-membrane pressure on propylene permeability for 70:30 (vol.%) propylene-propane mixture and at different carrier concentrations.

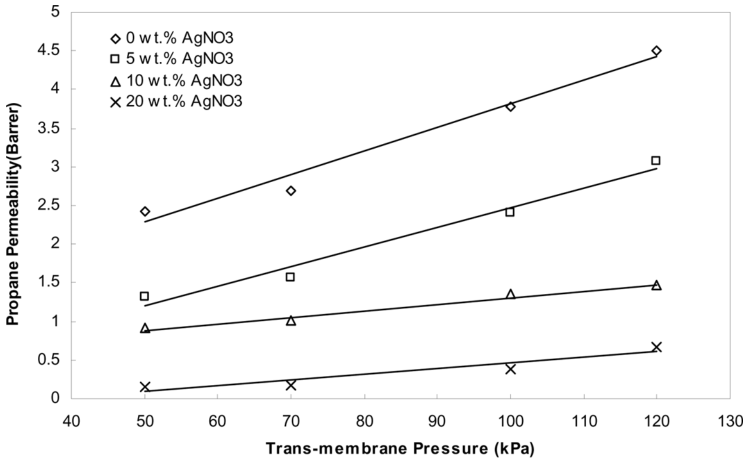

Figure 10. The effect of trans-membrane pressure on propane permeability for 30:70 (vol.%) propylene-propane mixture and at different carrier concentrations.

In facilitated transport membranes, propylene permeation occurs via two mechanisms: Fickian diffusion and facilitation transport. In the absence of carrier (Ag^+) in the membrane, propylene was permeated only via Fickian diffusion. When carrier was added to the membrane system, propylene was permeated via Fickian diffusion and facilitated transport.

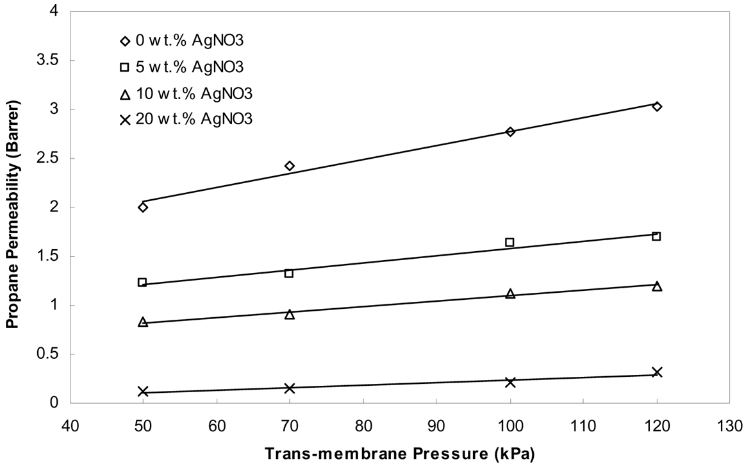

Figure 11. The effect of trans-membrane pressure on propane permeability for 50:50 (vol.%) propylene-propane mixture and at different carrier concentrations.

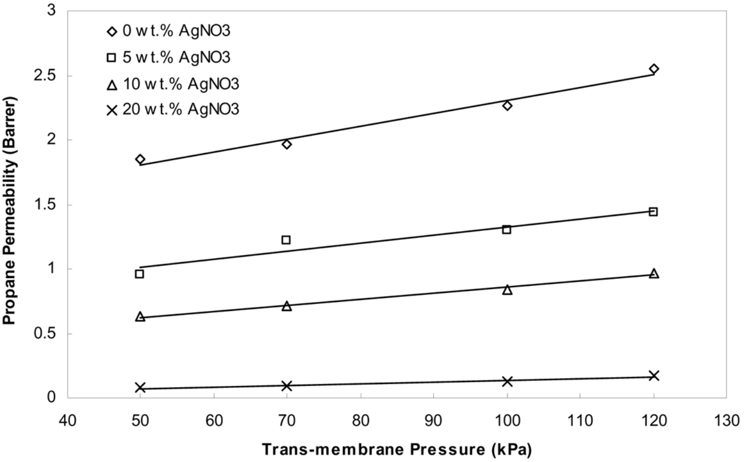

Figure 12. The effect of trans-membrane pressure on propane permeability for 70:30 (vol.%) propylene-propane mixture and at different carrier concentrations.

Based upon facilitated transport mechanism, when more carriers are available in the membrane, more propylene can be transported along the membrane thickness and this will cause an increase in the separation factor.

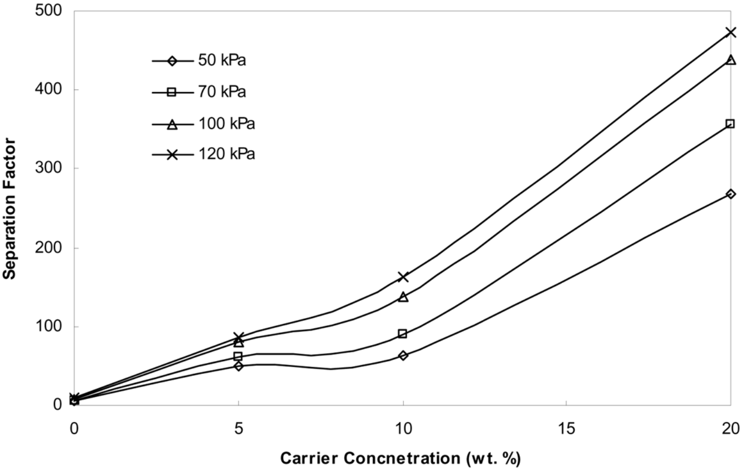

Figure 13. The effect of carrier concentration on the separation factor for 30:70 (vol.%) propylene-propane mixture and at different trans-membrane pressures.

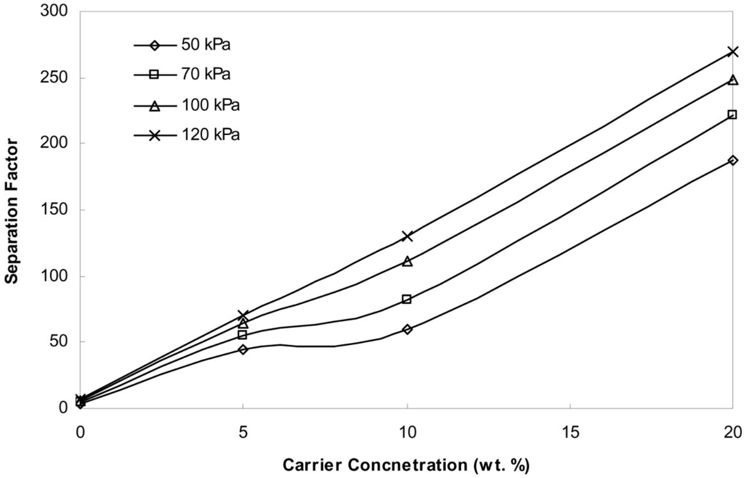

Figure 14. The effect of carrier concentration on separation factor for 50:50 (vol.%) propylene-propane mixture and at different trans-membrane pressures.

Effect of Propylene Partial Pressure

The effect of propylene partial pressure on membrane performance is shown in Fig. 16. As it can be seen, propylene partial pressure and propylene permeability are directly proportional.

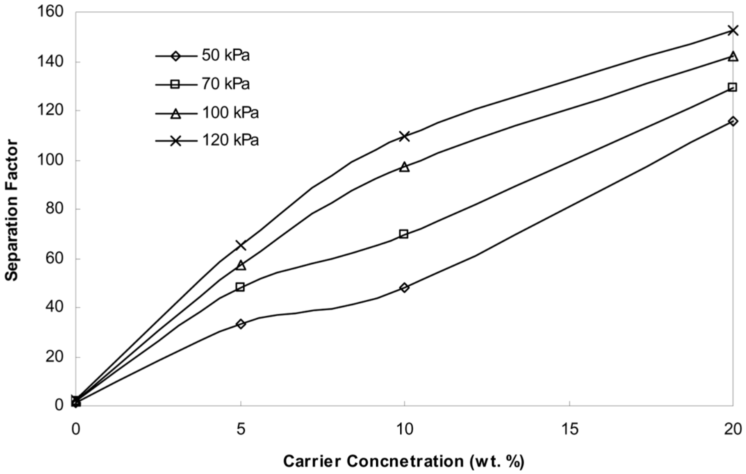

Figure 15. The effect of carrier concentration on separation factor for 70:30 (vol.%) propylene-propane mixture and at different trans-membrane pressures.

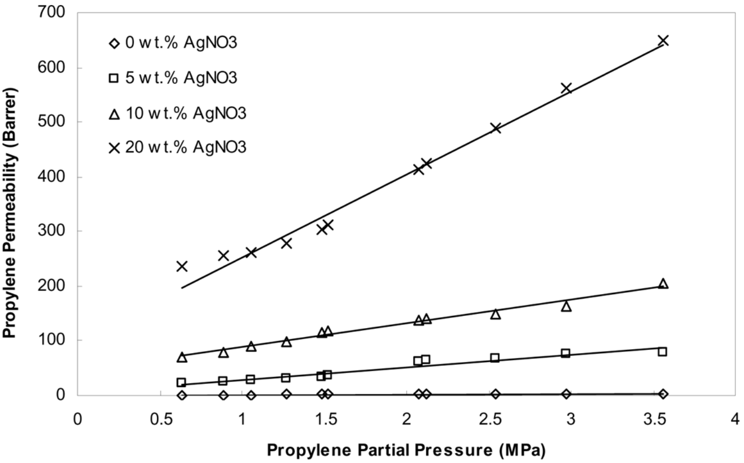

Figure 16. The effect of propylene partial pressure on propylene permeability at different carrier concentrations.

With increase in propylene partial pressure, the concentration gradient or the pressure gradient across the membrane thickness increases, i.e., the driving force in the membrane increases. Due to this increase in driving force, more propylene permeated across the membrane and propylene permeability increases.

Propylene permeability directly increases with carrier concentration. The more the carrier concentration in the membrane, the more the mass transfer and permeability of propylene.

Comparison between Facilitated Transport Membrane Systems for Olefin-Paraffin Systems

The performance of various facilitated transport membrane systems (solid polymer electrolyte ones) used for the separation of a 50:50 (vol.%)) propylene-propane mixture were summarized in Table 2. The highest separation factor obtained was 170 which was in PAAm-$AgBF_4$ membrane containing 67 mol.% Ag^+.

In the present work, with the use of an immobilized liquid membrane containing 20 wt.% Ag^+ for the separation of 50:50 (vol.%) propylene-propane mixture, a separation factor of 270 was obtained which is so much higher than the previous results.

Table 4 compared the performance of the present liquid membrane system with those reported in literature. The current immobilized liquid membrane system performs the best in comparison with other available

Table 4. A comparison between various membrane systems for olefin-paraffin separation

Membrane system	Olefin-paraffin	Separation factor	Reference
20 wt.% $AgNO_3$ aqueous solution	Propylene-propane[a]	270	Present work
PAAm-$AgBF_4$	Propylene-propane[a]	170	30
10 M $AgNO_3$ aqueous solution	Ethylene-ethane	1000	22
Nafion-6 M $AgBF_4$	Ethylene-ethane	30	41
PEO-$AgBF_4$	Ethylene-ethane	120	24
7.5 M $AgNO_3$-Glycerol	1-butene-n-butane	850	42
PVA- $AgNO_3$	Mixture of butanes and n-butane	96	39

[a] Feed mixture: 50 vol.% propylene, 50 vol.% propane

membrane configurations in terms of separation factor for propylene-propane separation.

A 10 M silver nitrate liquid membrane used for ethylene-ethane separation showed the highest separation factor. The carrier solution used in this system is more concentrated. Moreover, the mass transfer coefficient for a smaller molecule of ethylene is higher than that of propylene. These facts caused the separation factor to be higher.

Similarly, a 7.5 M silver nitrate-glycerol liquid membrane used for 1-butene-n-butane separation showed higher separation factor in comparison with the present work. This is because of the more concentrated carrier solution and different solvent used.

CONCLUSION

A series of experiments were carried out for the separation of propylene-propane mixtures using immobilized liquid membrane containing aqueous solution of silver nitrate ($AgNO_3$). The influence of trans-membrane pressure, carrier concentration, and propylene partial pressure on membrane performance was investigated.

It was found that increasing the trans-membrane pressure and carrier concentration is in favor of the separation factor and a more purified product was obtained. Moreover, the more the propylene partial pressure, the more the propylene permeability.

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NOMENCLATURE

L	Membrane thickness (cm)
N_i	Volumetric permeation rate of component i at standard pressure and temperature (cm^3 (STP)/s)
Q_i	Permeability (cm^3 (STP).cm/cm^2.s.cmHg, Barrer, 1Barrer $= 10^{-10}$ cm^3 (STP). cm)/cm^2.s.cmHg)
S_t	Total exposed membrane area (cm^2)
y	Mole fraction
ΔP_i	Partial pressure difference of component i across the membrane (cmHg)
α	Separation factor

Subscript

A	Propylene
B	Propane

Superscript

p	Permeate stream
f	Feed stream

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