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Gas Permeation Through Poly(Ether-*b*-amide) (PEBAX 2533) Block Copolymer Membranes

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ABSTRACT

The development of polymeric membranes for gas separations provides an alternative to traditional energy intensive processes, especially for hydrocarbon separations. We investigated the permeation behavior of several gases through poly(ether-*b*-amide) PEBAX 2533 copolymer membranes over a range of operating temperatures and feed pressures. Permeability coefficients were determined at temperatures ranging from 25°C to 75°C, and feed pressures from 25 to 200 psig for ethane, ethylene, nitrogen, propane, and propylene. Carbon dioxide was also studied to better appreciate the interaction between a polar gas and the membrane. The PEBAX 2533 membranes showed high hydrocarbon permeabilities. Plasticization effects on the membrane were pronounced with propane

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and propylene at elevated pressures. Activation energies of permeation were determined, which showed a linear decreasing trend with pressure increases for hydrocarbons.

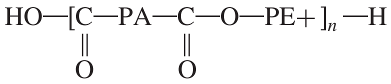
Key Words: Poly(ether-*b*-amide); Gas permeation; Operating temperature; Feed pressure.

INTRODUCTION

Over the past two decades, gas separation using polymeric membranes has drawn a great deal of interest from researchers due to its many advantages, such as low energy costs and high selectivities. This is especially true for hydrocarbon separations performed by the petrochemical industry where the separations of hydrocarbons incur a heavy cost to petrochemical companies.

The majority of industrial membrane processes for gas separations utilize glassy polymeric membranes because of their high permselectivity and good mechanical properties. Glassy polymers such as polyimides, are used for CO_2/CH_4 separation; polysulfones are used in H_2 separations; and cellulose acetate membranes are used for the removal of CO_2 and H_2S from natural gas. In the area of rubbery polymers, polyurethanes possess high permeability and are being applied in O_2/N_2 separation. Silicone polymers, particularly polydimethylsiloxanes (PDMS), are widely studied due to their large free volume and, hence, high permeability. Stern[1] presented a thorough review on the structure/permeability/selectivity relationships of selected rubbery and glassy polymers.

Polyether block amide (PEBA) resin is best known under the trademark PEBAX, and is a thermoplastic elastomer combining linear chains of rigid polyamide segments interspaced with flexible polyether segments. The history of PEBA resin can be dated back to 1972 when Atochem initiated research for a "soft nylon" material. The actual PEBA polymer was commercialized in 1981.[2] It is produced by polycondensation of a dicarboxylic polyamide and a polyether diol in the presence of heat, vacuum, and a catalyst.[3] The structure of the PEBAX repeating unit is:



$$\mathrm{HO{-}[\underset{\underset{\displaystyle O}{\|}}{C}{-}PA{-}\underset{\underset{\displaystyle O}{\|}}{C}{-}O{-}PE{+}]_{\mathit{n}}{-}H}$$

where PA is an aliphatic polyamide "hard segment" (i.e., Nylon-6 [PA6] or Nylon-12 [PA12]) and PE is an amorphous polyether "soft segment." The soft segment is either poly(ethylene oxide) or poly(tetramethylene oxide). This crystalline/amorphous structure creates a blend of properties of thermoplastics and rubbers. In application to permeation, it was believed that the hard amide block provides the mechanical strength, whereas gas transport occurs primarily in the soft ether segments.[4]

Currently, the main commercial applications of PEBA polymers range from sporting goods (shoe soles) and industrial equipment (conveyor belts) to functional films (breathable clothing, drying films) and various other materials. Only in recent years have membranes based on PEBAX polymer been investigated in separation processes. Rezac et al.[5] evaluated the sorption and diffusion characteristics of water and methanol in a series of PEBAX copolymers. Their results indicated that PEBAX materials could be used to selectively separate methanol from air, but not methanol from water. The PEBAX series in their study consists of Nylon-12 and polytetramethylene oxide of varying ratios. Among the four grades of PEBAX (2533, 3533, 5533, and 6333) studied, the 2533 grade was the most promising due to high permeation rates.

In applications involving the removal of CO_2 from gas mixtures, PEBA was found to have high selectivity for polar or quadrupolar/nonpolar systems (e.g., CO_2/H_2 or CO_2/N_2). Bondar et al.[4] evaluated membranes made from four grades of PEBAX, which range from 53 to 80 wt% of polyether (PE) in the block copolymers. The PEBAX grades used were 2533, 4011, 1074, and 4033 in descending PE weight percent. Their sorption and permeation results suggested strong interactions between the polar gas CO_2 and the PE blocks in the copolymers. It was found that PE composition and CO_2 permeability were directly correlated. Kim et al.[6] reported that the particular selectivity of CO_2 over N_2 is 61 and that of SO_2 over N_2 is 500. Strong affinity of polar species to the PE block is attributed to the high permeability and permselectivity of polar gases through PEBAX copolymer.

Although PEBAX copolymer has an excellent selectivity for organic/water separation in pervaporation[7,8] and for the separation of polar/nonpolar gas mixtures, there has been little work on the permeation of hydrocarbons through PEBAX membranes until a recent article by Morisato et al.,[9] who utilized membranes based on PEBAX 2533 doped with $AgBF_4$ to separate ethylene and ethane. The group reported the value of mixed-gas ethylene/ethane selectivity to be less than 2 as the $AgBF_4$ concentration increased from 0 to 70 wt% of the polymer mixture, while the selectivity increased to 26 when the salt concentration was raised from 70 to 90 wt%.

The objective of this study was to investigate the gas permeation properties of the solid poly(ether-*b*-amide) copolymer membrane PEBAX 2533 and to study the effect of operating conditions, temperature, and feed pressure on the gas permeation properties. Interactions between the organic permeant and the membrane material over a range of temperatures and pressures are also tested and discussed.

BACKGROUND

Solution-diffusion separation, which is based on both solubility and mobility factors, is the most commonly used model in describing gas transport in nonporous membranes. Koros et al.[10] gave a thorough review on polymeric membranes for solution-diffusion based permeation separations. Among the solution-diffusion-desorption stages, the diffusion step is the slowest; hence, it is the rate-determining step in permeation. The relationship between permeation flux and driving force is given by Fick's first law. When the solubility of a penetrant gas in a polymer is sufficient low, the concentration of the penetrant is proportional to the pressure of the penetrant, as expressed by Henry's law.

If Henry's law applies, then S, the solubility coefficient, is constant at a given temperature. When the diffusivity is independent of permeant concentration, the permeability coefficient, P, can be expressed as:

$$P = D * S \tag{1}$$

The diffusion coefficient, D, is a kinetic term governed by the amount of energy necessary for a particular penetrant to execute a diffusive jump through the polymer and the intrinsic degree of segmental packing in the matrix. The solubility coefficient, S, is a thermodynamic term that depends on factors such as condensibility of the penetrant, interactions between the polymer and penetrant, and in the case of glassy polymers, the amount of penetrant-scale nonequilibrium excess volume.

At steady state, and when downstream permeate pressure is much less than upstream pressure, the permeation of a pure gas A through a membrane of thickness x is characterized by a permeability coefficient P, which is generally defined as:

$$J = P\frac{p_1 - p_2}{x} = P\frac{\Delta p}{x} \tag{2}$$

where p_1 and p_2 are the upstream and downstream pressures, respectively, Δp is the pressure difference $(p_1 - p_2)$ across the membrane, and J is the permeation flux.

For a binary gas mixture permeating through a polymer membrane, the selectivity of a polymer membrane toward two different penetrant gases, A and B, is commonly expressed in terms of the ideal selectivity or ideal permselectivity, α_{AB}. When the downstream pressure is negligible relative to the upstream pressure, α_{AB} can be written as the ratio of permeabilities:

$$\alpha_{AB} = \frac{P_A}{P_B} = \left(\frac{D_A}{D_B}\right)\left(\frac{S_A}{S_B}\right) \tag{3}$$

In typical gas separation applications, the downstream pressure is not negligible; however, α_{AB} generally provides a convenient measure for assessing the relative ability of various polymers to separate gas mixtures by permeation. High permeability and high selectivity are the most important criteria in selecting and evaluating a membrane.

EXPERIMENTAL

Membrane Preparation

The PEBAX (grade 2533) copolymer was supplied by ATOFINA Chemical, Inc. (Philadelphia, PA). PEBAX 2533 flat film (area = 9.6 cm^2, average thickness = 20 μm) was prepared by the solution casting method. 1-Propanol, certified ACS grade, was used to make a 3 wt% PEBAX 2533 solution. The 3 wt% polymer concentration was decided based on viscosity considerations. The polymer was dissolved at 90°C while vigorously stirring over a 48-hour span and found adequate for complete dissolution. The polymer solution was poured into a Petri dish and dried at room temperature for 48 hours under a fume hood such that solvent was completely removed.

Dried membranes were first cut into desired configurations, placed on a support material, and subsequently transferred to the membrane cell for permeation tests. Nonwoven polyester fabric was used as the support material to increase the mechanical strength of the membranes during permeability tests. Gas permeation through the support fabric was negligible since gas resistance in the fabric support is much smaller compared to that in the membranes.

Permeation Tests

Membrane permeability tests were performed using five gases (nitrogen, ethane, ethylene, propane, and propylene). Selectivities (e.g., in Table 1 and Fig. 6) were evaluated using pure gas permeation ratios. In addition, the permeability of carbon dioxide was studied to evaluate the selectivity of PEBAX with respect to a polar system. All gases were purchased from Praxair Inc., with at least 99% purity. The feed pressure ranged from 25 to 200 psig, while the permeate pressure was maintained at atmospheric (0 psig).

Table 1. The effect of feed pressure on selectivities of different gas pairs (at 25°C).

	Selectivity			
Pressure (psig)	C_3H_6/C_3H_8	C_2H_4/C_2H_6	C_3H_8/C_2H_6	C_3H_6/C_2H_4
25	1.6	1.4	3.5	4.0
50	1.8	1.4	4.4	5.8
75	2.0	1.3	5.4	8.0
100	2.0	1.2	7.5	11.9

	Selectivity			
Pressure (psig)	C_3H_8/N_2	C_3H_6/N_2	C_2H_6/N_2	C_2H_4/N_2
25	26.4	43.1	7.6	10.7
50	36.8	65.1	8.3	11.2
75	48.0	94.7	8.8	11.8
100	77.3	152.5	10.3	12.9

The operating temperature varied from 25°C to 75°C. The permeation cell was placed in a 6-L VWR thermal bath (Model 1167) with a built-in temperature controller. The experimental set-up and the structure of the permeation cell can be found in an earlier publication.[11] Prior to each permeation experiment, the membranes were "conditioned" at two or three times the normal testing pressure.

RESULTS AND DISCUSSION

Effect of Feed Pressure

The permeabilities of PEBAX 2533 film were determined as a function of feed pressure. A change in the pressure of penetrant contacting the polymer may cause large permeability variations. Four typical patterns of response are usually observed in a permeability vs. pressure relationship:[12]

1. A slope close to zero represents the (ideal) case where both diffusivity and solubility coefficients are not significantly affected by gas pressure (i.e., low sorbing penetrants, such as He or N_2, in rubbery or glassy polymers).
2. Nearly linear increase of permeability with increasing pressure. This often describes the permeability of an organic vapor into a rubbery polymer.

3. A decreasing trend of permeability with increasing pressure. This is typically observed with highly soluble gases, such as CO_2, in glassy polymers.
4. Concave upward. This can be perceived as a combination of (2) and (3), and is typical of a plasticizing penetrant, such as organic vapor, in a glassy polymer.

Representative permeability data for ethane, propane, and nitrogen are shown in Fig. 1, and for ethylene and propylene in Fig. 2. The permeabilities of ethane and ethylene have an almost linear relationship to feed pressure, which conforms to (2) above. The curved nature of the responses from propane and propylene indicates the plasticization effect [see (4) above]. The permeability of nitrogen is not significantly affected by feed pressure under the experimental conditions, representing case (1).

At higher pressure, the permeabilities of propane and propylene strongly affect the selectivities for gas pairs involving propane and propylene. Table 1 presents the calculated selectivities of different gas pairs in relationship with pressure changes. It can be seen that selectivity tends to increase with an increase in feed pressure for the pairs of propylene/ethylene, propane/ethane,

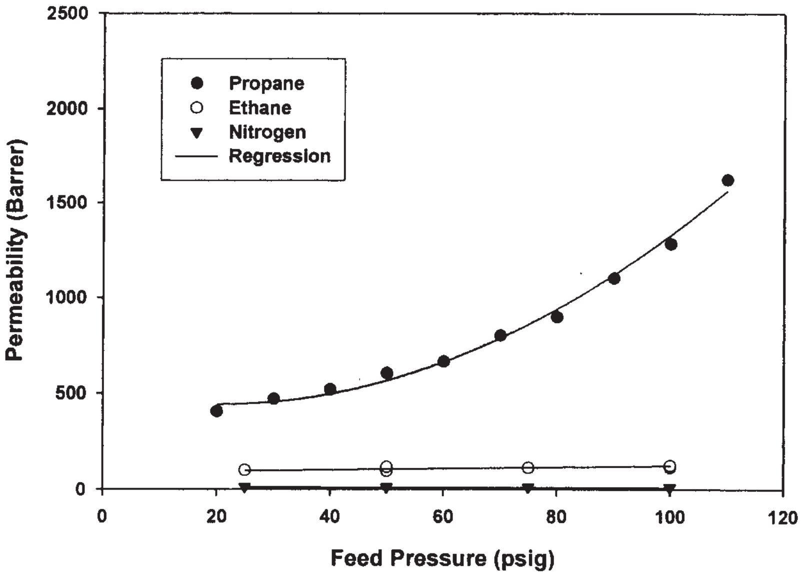


Figure 1. Permeability as a function of feed pressure for permeation of nitrogen, ethane, and propane through PEBAX 2533 membrane at 25°C.

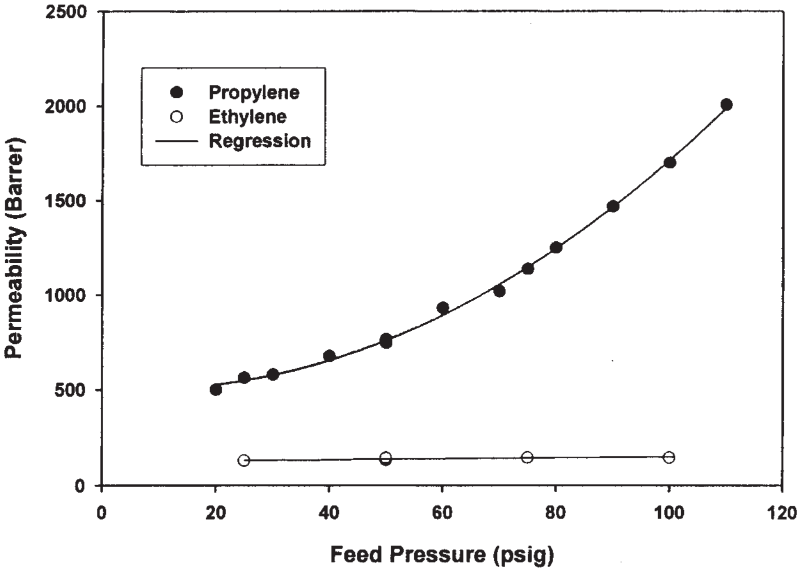


Figure 2. Permeability as a function of feed pressure for permeation of ethylene and propylene through PEBAX 2533 membrane at 25°C.

propane/nitrogen, and propylene/nitrogen. For the ethane/nitrogen and ethylene/nitrogen pairs, there is a slight decrease in selectivities when feed pressure is increased. This may be explained from Eq. (3). Although gas diffusivity selectivity (D_A/D_B) increases as pressure increases, the solubility selectivity (S_A/S_B) decreases, causing a counter effect and thus reducing the overall permselectivity. Selectivities remain almost constant for the ethane/ethylene and propane/propylene pairs, since pressure has similar magnitude effects on both components (D_A/D_B) and (S_A/S_B) of Eq. (3).

Effect of Temperature

The effect of temperature on D, S, and P can be described by the Arrhenius relationships:

$$D = D_0 e^{-E_d/RT} \tag{4}$$

$$S = S_0 e^{-\Delta H_s/RT} \tag{5}$$

$$P = P_0 e^{-E_p/RT} \tag{6}$$

where E_d is the activation energy of diffusion; ΔH_s is the heat of sorption; and

$$E_p = E_d + \Delta H_s \tag{7}$$

is the activation energy of permeation.

Gas diffusion coefficients typically increase appreciably with increasing temperature when the polymer does not undergo thermally induced morphological rearrangements, such as crystallization, over the temperature range of interest.[13] Since both diffusivity and solubility coefficients are temperature dependent, selectivity is also sensitive to changes in temperature.

The increased segmental motion at higher temperatures undermines the ability of polymer to discriminate between penetrants of different physical dimensions, thereby resulting in a diffusivity selectivity loss. The temperature changes also affect the solubility selectivity, which is governed primarily by the chemical nature of the penetrant and polymer-penetrant interactions. For most gases, as temperature increases, solubilities decrease. The solubility selectivity, therefore, will vary depending on the extent of the temperature effect on each component in the gas mixture.[14]

Figure 3 shows the temperature dependence of propane permeation at different feed pressures. On a semi-log graph, the relationship between permeability and inverse temperature is linear. A linear regression was applied

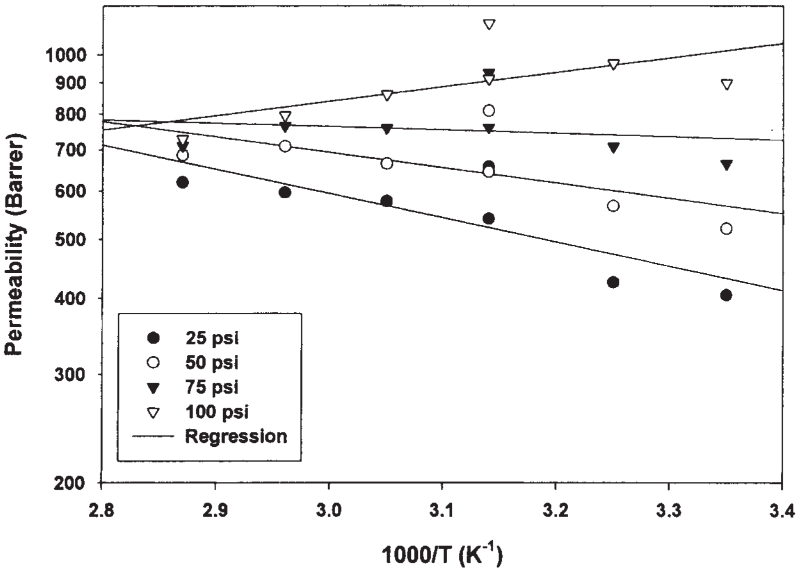


Figure 3. Temperature dependence of propane permeability in PEBAX 2533.

to the permeability data of those gases tested. Pre-exponential values and the apparent activation energy of permeation were determined and are summarized in Table 2, which clearly shows that as pressure increases, the activation energy of propane permeation decreases. Similar trends were also observed for propylene permeation. The negative entries for E_p can be explained via Eq. (7) and the relative magnitudes of E_d (positive) and ΔH_s (negative).

Nitrogen shows a distinctly different trend from propane and propylene. Figure 4 shows that all regression lines are similar in slope and position. Nitrogen has higher pre-exponential and activation energy values, which are also presented in Table 2. Ethane and ethylene exhibited similar behavior to nitrogen, but there is a slight decrease in activation energy as pressure increases, as also shown in Fig. 5. This indicates that the permeability of nitrogen in PEBAX is not significantly affected by temperature changes and that C3 hydrocarbons are more affected by temperature than C2 hydrocarbons are.

Table 2. Calculated values of pre-exponential factors and activation energy.

	Propane		Propylene	
Pressure (psig)	P_0 (Barrer)	E_p (KJ/mol)	P_0 (Barrer)	E_p (KJ/mol)
25	1.0E+04	8.0	5.3E+04	11.4
50	4.4E+03	5.2	1.9E+04	8.1
75	1.3E+03	1.5	8.5E+02	−0.7
100	1.9E+02	−4.1	9.1E+01	−7.1
	Ethane		Ethylene	
	P_0 (Barrer)	E_p (KJ/mol)	P_0 (Barrer)	E_p (KJ/mol)
25	4.4E+05	21.6	3.0E+05	19.2
50	1.9E+04	18.3	1.5E+05	17.2
75	1.9E+04	16.8	1.5E+05	17.2
100	1.6E+04	15.9	1.3E+05	16.7
	Nitrogen			
	P_0 (Barrer)	E_p (KJ/mol)		
25	1.6E+05	23.4		
50	2.1E+05	24.3		
75	9.7E+04	22.3		
100	1.9E+05	24.2		

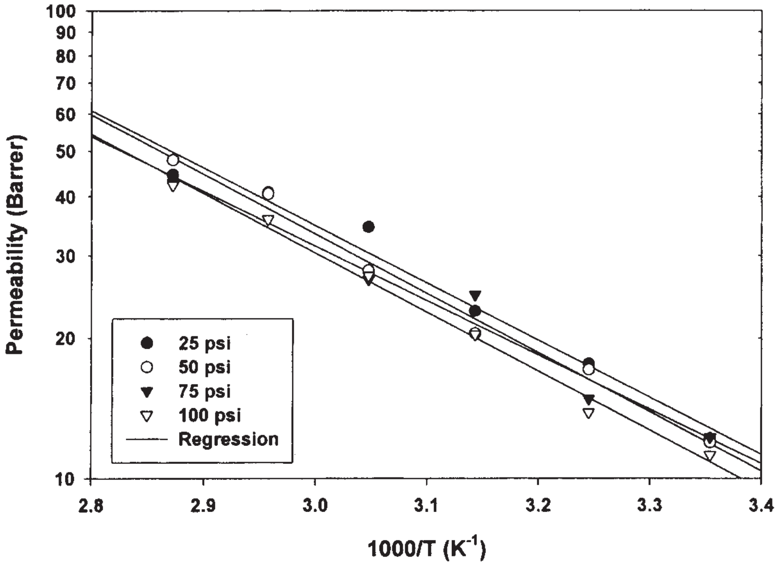


Figure 4. Temperature dependence of nitrogen permeability in PEBAX 2533.

Effect of Other Factors

Apart from the operating conditions (i.e., operating temperature and feed pressure), other factors such as composition (in the case of gas mixtures), penetrant condensibility, polymer-penetrant interactions, and polymer crystallinity may also affect gas solubility. Furthermore, gas diffusivity is sensitive to properties such as penetrant size, polymer morphology, and polymer segmental dynamics.

In general, gas solubility in polymers increases with increasing gas condensibility. The condensibility of a gas can be measured by its critical temperature T_c, or the normal boiling point T_b. Diffusion coefficients of penetrants are found to decrease with increasing penetrant size. Diffusion coefficients in polymers are also sensitive to penetrant shape. Linear or oblong penetrant molecules like CO_2 exhibit higher diffusivities than those of spherical molecular shape of equivalent molecular volume such as CH_4. A summary of T_c and kinetic diameter values for the studied gases can be found in Table 3. Specific interactions between gas and polymer molecules (i.e., polarity) also affect gas solubility. Gases such as CO_2, which has a quadrupole moment, are generally more soluble in polar polymers. In the permeation studies done by Kim et al.,[6] with PEBAX 1657 at a pressure of 3 atm,

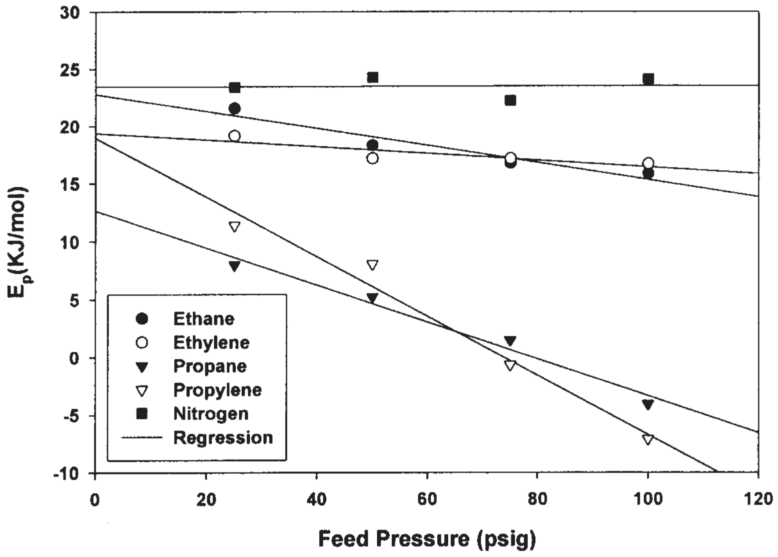


Figure 5. Pressure dependence of activation energy of permeation.

selectivity of CO_2/N_2 exhibits inverse temperature dependence. We have confirmed a similar trend using PEBAX 2533 at 25 psig and temperatures from 25°C to 75°C (see Fig. 6).

Plasticization may occur at a pressure where the gas concentration in the polymer material disrupts the chain packing. The polymer matrix swells and the segmental mobility of the polymer chains increases. This results in an increase in gas diffusivity and induces permeability increases.[17] Therefore,

Table 3. Summary of critical temperature and kinetic diameter values for the gases studied.

Gases	T_c (K)[a]	Kinetic diameter (nm)[b]
N_2	126.20	0.36
CO_2	304.21	0.33
C_2H_6	305.32	—
C_2H_4	282.34	0.39
C_3H_8	369.83	0.43
C_3H_6	365.57	0.45

[a] Perry's Chemical Engineering Handbook, 1999.[15]
[b] Breck, 1974.[16]

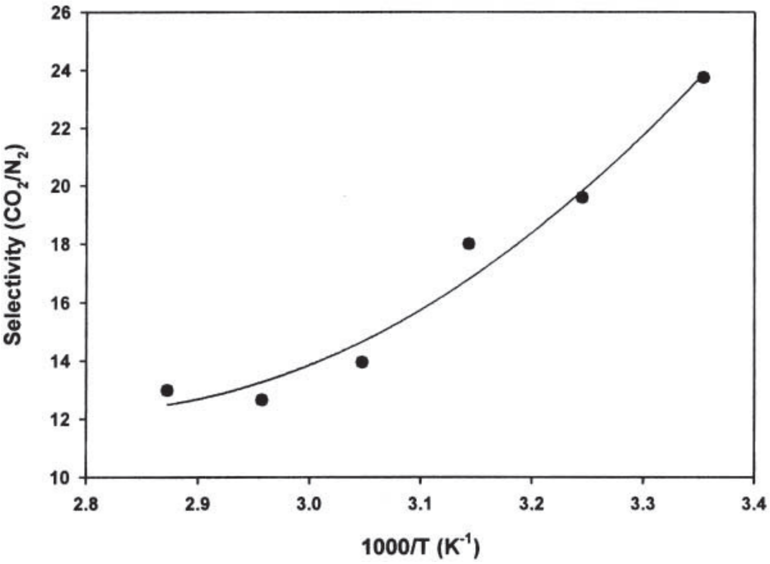


Figure 6. Temperature dependence of CO_2/N_2 selectivity for PEBAX 2533 at 25 psig.

when the polymer is highly plasticized by the penetrant, the diffusion coefficient may become a function of time and history. It has been observed that PEBAX retains the "memory" of prior permeations. After a set of permeation tests was run under various temperatures, another set of permeation tests was preformed at 25°C. For propane gas, the second set of tests showed lower permeability than the first time, as shown in Fig. 7. This indicates that plasticization had taken place with temperature and pressure changes, and the PEBAX structure was altered. Similar results were observed with the other hydrocarbons tested in our studies. Certainly, more detailed studies are required to investigate and clarify further the "history" effect.

CONCLUSION

PEBAX 2533 membranes showed high hydrocarbon permeability. The permeability of PEBAX 2533 film was determined as a function of feed pressures and operating temperatures. Permeation properties obtained with propane and propylene indicated strong plasticization effects on the PEBAX 2533 polymer matrix. Nitrogen showed little interaction with the membrane material, which is considered ideal. Permeabilities of propane and propylene are

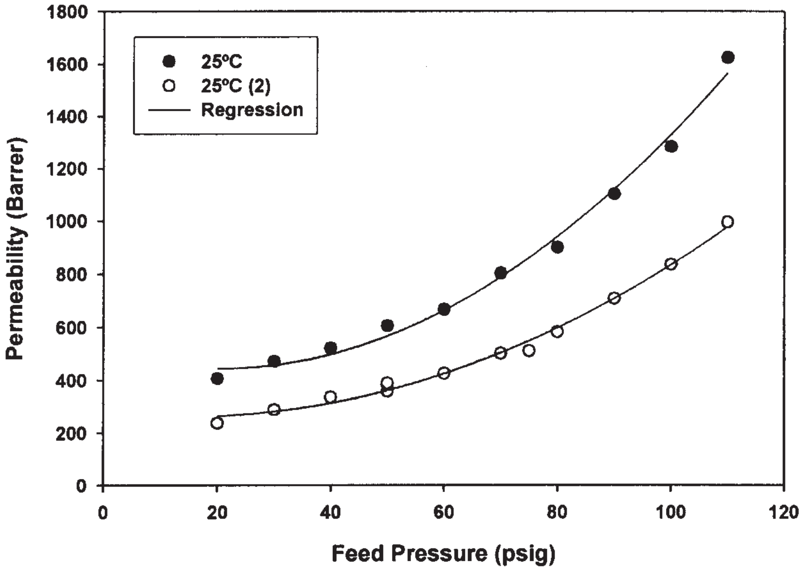


Figure 7. Permeability as a function of feed pressure. Propane through PEBAX 2533.

strongly affected by both operating temperature and feed pressure. As pressure increases, the permeability of propane and propylene increases. The temperature dependence of permeability can be approximated by Arrehnius expressions. The activation energy of permeation decreases as pressure increases. Due to the polar nature of the membrane material, it has been investigated for separating polar and nonpolar gas pairs. CO_2/N_2 separation is temperature sensitive with PEBAX and higher selectivity is achieved at lower temperature.

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