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Publisher's version / Version de l'éditeur:

<https://doi.org/10.1016/j.memsci.2010.05.059>

Journal of membrane science, 361, September 1-2, pp. 176-181, 2010

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Gas separation transport modeling for PDMS coatings on PEI-PEG IPN membranes[☆]

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ARTICLE INFO

Article history:

Received 31 March 2010

Received in revised form 25 May 2010

Accepted 28 May 2010

Available online 4 June 2010

Keywords:

Gas separation

Bi-layer system

CFD

Selectivity

Modeling

Simulation

ABSTRACT

A caulking layer is frequently applied to gas separation membranes to alleviate the effects of structural defects. Caulking materials have high gas diffusivities, so as not to hinder the performance of the gas separation layer. Despite these considerations, for some bi-layer systems unaffected by plasticization, the mixed gas selectivity has been observed to be significantly lower than selectivity values calculated from pure gas permeabilities. The present object is to model and simulate two-layer caulked gas separation membranes at the fundamental level to discover the mechanistic basis for the observed behaviour.

To this end, a transport based model considering intrinsic material properties was developed to simulate gas separation for a system of CO₂ and N₂ diffusing across a caulking layer of polydimethyl siloxane (PDMS) and then a polyetherimide-polyethylene glycol interpenetrating network (PEI-PEG IPN) membrane. The model was able to account for flux declines in mixed gas systems greater than those predicted by conventional series resistance models, such as have been observed experimentally.

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1. Introduction

The use of polydimethyl siloxane (PDMS) as a caulking material on non-defect free asymmetric polymer membranes for gas separations is widespread [1,2]. Resistance models can show that at large thickness values, the pure gas selectivity of a caulked membrane can take on the characteristics of the caulking material rather than those of the membrane material [3]. It has been shown experimentally and via modeling that a range of PDMS coating thicknesses can result in pure gas selectivities which approach those of the membrane material, while showing mixed gas selectivities more typical of the PDMS coating. In cases where the permeance of the PDMS coating is significantly less than the membrane, the pure gas selectivity does not appear to be affected. However, with mixed gases, the presence of the PDMS layer alters the gas composition at its interface with the membrane, thus influencing the net observed selectivity.

Examples can be found in the literature of modeling the effect of the support resistance on permeation [4–8]. A more thorough analysis can be performed by using a fundamental transport approach, and simulating the overall permeance through considering the independent effects of diffusion and solubility. A transient computational fluid dynamics (CFD) model was developed here to

demonstrate the effects and consequences of the proposed model through its illustration of the system behaviour.

2. Materials and experimental

The details regarding preparation of PEI-PEG IPNs were reported by Saimani and Kumar [9].

The IPN membranes were coated with 0.5, 1.0 or 3 w/v% PDMS in pentane (Sylguard), by flushing the surface of the vertically inclined membrane with solution. The bottom of the membrane was in contact with a surface to facilitate draining of the solution. The membrane was allowed to dry at room temperature before application of subsequent coatings of the PDMS solution.

Membrane samples were tested for pure and mixed gas permeability using the test cells described in [10].

Gases were obtained from BOC (Ottawa, Canada), the air was dry and CO₂ free grade, O₂ and N₂ were ultra-high purity or equivalent, and the CO₂ was humidity free. Molar feed ratios of CO₂/N₂ were prepared at 10, 20, 30 and 40% using MKS 180A ALTA (Andover, MA, USA) mass flow controllers. Feed and permeate concentrations for CO₂ were measured using a Quantek 906 (Grafton, MA, USA) CO₂ analyzer with a 0–100% range. Oxygen concentrations for air permeation experiments were measured using an Advanced Micro Instruments O₂ meter, Model 65 (Garden Grove, CA, USA), calibrated to 20.9% oxygen for air with a range of 0–95%.

Permeate flows were measured using an ADM 1000 flowmeter (Agilent Technologies, Mississauga, Canada), with a range of

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Fig. 1. Selectivity (CO_2/N_2) as a function of CO_2 permeance. Open symbols indicate the pure gas data, dashes are for mixed gas data for the material system indicated by each colour.

$0.5\text{--}1000\text{ ml min}^{-1}$, reporting flows in ml min^{-1} at 25°C . Permeate flows below 1 SCCM were measured using the MKS mass flow controller. The permeance was corrected to standard temperature and pressure (STP) from a typical room temperature of 22°C .

Membranes were initially evaluated using air and allowed to stabilize overnight. The pure gas permeance was measured in the sequence N_2 , O_2 , CO_2 , N_2 at 689,010 Pa (6.8 atm) (100 psig). Mixtures were measured in increasing CO_2 feed concentrations and the pure gas sequence repeated in selected cases. Feed flows with mixed gases were maintained at 1500 ml min^{-1} and permeate recovery was generally maintained below 2%.

In Fig. 1 are some experimental data showing selectivity declines for various PEI-PEG IPN formulations with PDMS caulking layers. The different mixed gas data points are for different CO_2 feed concentrations. The main point to be made here is that the data show a general selectivity and flux decline for mixed-gas cases compared to pure gas cases.

Film thickness measurements were not made for these tests, but the PDMS layers were known to be significantly thicker than the membrane layer. At the time of these experiments, the present study had not been conceived, and the work was being done with different aims. The curious data however, posed some questions on the nature of the system behaviour, and gave rise to the analysis undertaken in this paper. Given that the experiments were conducted without a view to the objectives expressed here, and in the interests of communicating new theoretical results, they are presented despite the fact that only partial relevant data were recorded. In any event, these data serve to illustrate that feed composition dependent selectivity declines occur in mixed gas separation systems with thick caulking layers applied.

3. Theory

3.1. Gas separation fundamentals

The transport of gases through a membrane is described by one dimensional Fickian diffusion:

$$\frac{\partial c}{\partial t} = D \frac{\partial^2 c}{\partial x^2} \quad (1)$$

Above, c is the gas concentration, D is the diffusivity, t is time, and x represents a spatial dimension.

At steady state, the flux J , of a gas species becomes uniform across a membrane or membrane layers, and is given by:

$$J_{ij} = D_{ij} \frac{\Delta c}{\Delta x} \quad (2)$$

The concentration boundary condition is:

$$c_{ij} = f_j P_{\text{FEED}} S_{ij} \exp \left[\frac{v_j (P_{\text{FEED}} - P_{\text{SAT},j})}{RT} \right] \quad (3)$$

Above, for gas species j , in material i , S is solubility, P is pressure, f is feed mole fraction and v is the partial molar volume of the species j dissolved in the membrane material, considered to be approximately equal to the molar volume of liquid j . $P_{\text{SAT},j}$ is the saturation vapour pressure of component j [11].

3.2. Bi-layer systems

For reference, we refer to a combined resistance model developed by Henis and Tripodi [3]. The Henis model assumes that the (two) gas species diffuse independently. The expression for permeation per unit film thickness is given in Eq. (4).

$$\frac{P_{j,\text{Total}}}{\Delta x_{\text{Total}}} = \left(\frac{\Delta x_1}{P_{j,1}} + \frac{\Delta x_2}{P_{j,2}} \right)^{-1} \quad (4)$$

In Eq. (4), P is the permeability, the indices 1 and 2 refer to the different material layers, and the index j refers to a particular species of permeating gas. It should be noted that the form of the Henis model given by Eq. (4) is for material layers which are defect free. Frequently the model is expressed in a form where one or more of the layers contains some surface fraction of defects, which are incorporated into it as a parallel resistance with an extremely high permeability. The application of the Henis model to explore the effects of material defects is not within the scope of the present work.

With the physical requirement of equal flux in both layers, the Henis model predicts overall permeation and selectivity reductions at large coating layer thicknesses. This is illustrated schematically in Fig. 2. The two cases illustrated with green lines attain the theoretical permeability. Note that over the caulking layer their slopes are the same as required by the flux definition based in $\Delta c/\Delta x$ and the value of the concentration (in mol m^{-3} for a single gas, linearly proportional to the pressure through the ideal gas law) at the caulking-membrane interface is sufficiently high to allow the same flux to occur through the membrane layer. As the caulking layer thickness continues to increase, the theoretical permeability applied across the caulking layer will produce a caulking layer-membrane interface concentration below the value that would allow the same flux to occur through the membrane. That is, the maximum $\Delta c/\Delta x$ available across the membrane represents a gas flux less than what the unconstrained permeability value for the gas-membrane pair would suggest. What results is depicted with the solid red line in Fig. 2, where a net bi-layer flux reduction

Fig. 2. Schematic of single gas flux reduction due to thick coating layer, as could be predicted by the Henis model.

Fig. 3. Schematic of permeation and selectivity reduction mechanisms in a bi-layer system.

moves the interfacial concentration to a higher value than would be obtained with theoretical permeability through the caulking layer, such that the ensuing slope through the membrane layer, when considered together with the different diffusivities through both materials, results in an equal, and reduced gas flux through both layers of the bi-layer system. In Fig. 2, the point C_{CRIT} shows the minimum concentration permitted on the product side of the PDMS layer which would still result in the limiting theoretical permeability to be obtained. PDMS layer thicknesses which require the product side concentration to be below C_{CRIT} will necessarily result in flux declines.

When the Henis model predicts mixed gas flux reductions across bi-layer systems, it does so only through considering the effects attributable to the different layer thicknesses, applied independently to each diffusing species of gas. As mentioned above, flux reductions greater than those predicted by the Henis equation have been observed for permeating mixed gas systems. Next, it is shown that a basic transport approach can incorporate the additional effect of local solubility limits, which also contribute to permeation and selectivity reductions.

Fig. 3 is a schematic depiction of the situation of two gases permeating across a caulked membrane. In a bi-layer case where theoretical permeation values are attained for both gases in a two gas situation, the Henis model will correctly predict the system selectivity, as it is only dependent on the material properties and dimensional characteristics of the two layers. In cases where a permeation or selectivity below that suggested by Eq. (4) is observed, it will be demonstrated that incorporating solubility effects into the transport model can produce a more coherent depiction of the system behaviour.

The case shown by the dashed lines in Fig. 3 represents a situation where the theoretical permeation rates for both gas species can be attained across the membrane. The gas permeation rate is normally lower in the membrane than in the caulking layer and represents a limiting permeation value. A deviation in the observed behaviour arises when the diffusing species concentration at the caulking layer–membrane interface reaches the solubility limit of the membrane layer. Feed side concentrations on the PDMS caulking layer are determined by Eq. (3). Imposing expected theoretical permeation rates based on the membrane properties (ie; the dashed lines in Fig. 3) suggests a feed side concentration of CO_2 greater than the solubility limit, depicted by the point in the green box. In reality of course, the saturation concentration is the max-

imum allowable species concentration. The concentrations at the caulking layer–membrane interface are also governed by Eq. (3), with the feed mole fraction determined from the species concentrations at the exit side of the caulking layer. The net consequence is that the feed side concentration of a gas species on the membrane is at times restricted by the solubility limit, thereby decreasing its flux across the membrane, and accordingly the observed system selectivity. This reduced permeation rate scenario is depicted with the solid lines in Fig. 3.

For the present case, the diffusivities of CO_2 and N_2 in PDMS make for a decreasing CO_2 mole fraction as the PDMS layer thickness (δ_{PDMS}) increases, which decreases c_{CO_2} at the PDMS–PEG interface as determined by Eq. (3). Further, c_{CO_2} and c_{N_2} on the PEG side respect saturation limits. The higher permeability of CO_2 in the PDMS caulking layer actually serves to decrease its selectivity against N_2 in a bi-layer system.

4. Modeling approach

An objective here is to employ only intrinsic properties such as diffusivity and solubility, in order to predict system performance parameters such as permeability and selectivity. Permeability is a derived system parameter, and is most meaningful for the transport of a single gas across a single material layer. As it is the aim of the present study to demonstrate how both layer thickness and solubility effects determine the net system transport behaviour, the concept of permeability will be referred to later in the analysis of the results, as a comparative measure of system performance.

The separation of CO_2 from N_2 is simulated, according to the configuration shown in Fig. 4. Table 1 summarizes the physical properties of the gas species considered in this work. Diffusivity and solubility parameters were taken from [12–14]. Parameter values for the partial molar values were found in [15–17]. Saturation vapour pressure values were taken from [18].

At steady state, determining the eight concentrations shown in Fig. 3 will resolve the system with Eqs. (1)–(3). At infinite time (steady state), the additional relation,

$$f_{CO_2,PERMEATE} = \frac{J_{CO_2}}{J_{CO_2} + J_{N_2}} \quad (5)$$

is employed to help resolve the system.

At the PDMS–PEG interface, the boundary condition for species concentration based on chemical potential (Eq. (3)) was used, since solubilization processes are considered to be more rapid than diffusional processes [11]. Expressing Eq. (3) explicitly for the

Fig. 4. Outline of material properties and operational parameters used in the bi-layer permeation and separation models.

Table 1

Physical properties of the materials and gases simulated in the bi-layer permeation study.

	Gas (at 35 °C)				
	P	S	D	ν	P_{SAT}
Layer material	$\text{mol s}^{-1} \text{ m}^{-2} \text{ Pa}^{-1}$	$\text{mol m}^{-3} \text{ Pa}^{-1}$	$\text{m}^2 \text{ s}^{-1}$	$\text{m}^3 \text{ mol}^{-1}$	Pa
CO ₂				5.36×10^{-5}	7.80×10^6
PEG	3.682×10^{-14}	5.72×10^{-4}	6.20×10^{-11}		
PDMS	1.517×10^{-12}	9.00×10^{-4}	1.68×10^{-9}		
O ₂				1.89×10^{-5}	5.53×10^6
PEG	2.678×10^{-15}	4.23×10^{-5}	5.38×10^{-11}		
PDMS	3.124×10^{-13}	1.50×10^{-4}	2.08×10^{-9}		
N ₂				2.11×10^{-5}	3.55×10^6
PEG	7.366×10^{-16}	2.45×10^{-5}	3.01×10^{-11}		
PDMS	1.473×10^{-13}	1.22×10^{-4}	1.21×10^{-9}		

PDMS–PEG interface,

$$c_{\text{PEG},j} = f_{\text{PDMS},j} P_{\text{MID}} S_{\text{PEG},j} \exp \left[\frac{v_j (P_{\text{MID}} - P_{\text{SAT},j})}{RT} \right] \quad (6)$$

The above expression also serves to couple to diffusional processes in the two material layers through the interfacial boundary condition. That is, the feed side concentrations and relative mole fractions are determined by the mole fractions at the exit side of the caulking layer. There is no such coupling component found in the Henis model.

When determining boundary concentrations for diffusion into the PEG layer, the saturation limits for two gas cases were determined by scaling according to mole fraction, expressed as,

$$c_{\text{SAT},j} = f_{\text{PEG},j} S_{\text{PEG},j} \quad (7)$$

5. Results and discussion

The system of steady state equations (Eqs. (1)–(3)) was solved analytically to produce selectivity results for CO₂ and N₂ dif-

fusing in a bi-layer PDMS–PEG system for a range of PDMS thicknesses.

The same calculations were also done with a transient CFD (Computational Fluid Dynamics) simulation, on a uniformly spaced one-dimensional mesh of 2000 cells. Grid refinement tests were run, and found that solution resolution no longer improved with finer computational meshes than this. The open source software OpenFOAM was used for these simulations [19]. For a boundary condition on permeation out of each layer, $\nabla(\nabla c) = 0$ was set. An initial time step of 10^{-10} s was used, and was progressively increased to 10^{-5} s over a total time period of 0.05 s, at which point steady state was always found to have been reached. The initial conditions were obtained by calculating the feed side concentrations at atmospheric pressure, then setting the CO₂ and N₂ concentrations equal to this inside the PDMS layer. At the PDMS–PEG interface, Eq. (6) was applied to determine the initial concentrations of CO₂ and N₂ in the PEG layer. At the start of the simulation, the feed side pressure was raised to the operating value of 7.9×10^5 Pa, which served to raise the feed side concentrations, and the simulation then ran over time for 10^5 iterations.

Fig. 5. Time series of concentration profiles for CO₂ and N₂ inside PDMS and PEG layers from initial state until steady state. The final plot shows data from both the equilibrated transient simulation with the results from the analytical steady state model. The case shown here is for a feed CO₂ mole fraction of 0.5 and $\delta_{\text{PDMS}}/\delta_{\text{PEG}} = 10$.

Fig. 6. Comparison of selectivity predictions from both the analytical transport model and the transient CFD model for the system of CO₂ and N₂ across a bi-layer PDMS-PEG system for a range of film thickness ratios and two feed concentrations against predictions from the Henis model.

The data from these solutions and simulations are shown in Figs. 5 and 6. Application of the PDMS-PEG interface boundary condition, Eq. (3), raised the point of the pressure distribution across the membrane layers. Classic gas separation models have treated this point in the following ways: The solution-diffusion model assumes that the pressure within a membrane is uniform with a constant concentration gradient [20]. The pore-flow model assumes that the pressure gradient is uniform, while the permeating species concentration is constant [21]. Neither of these representations is suitable for a basic transport approach which considers concentration profiles inside the membrane layers. Understandably, those models are suitable for a unit operation approach considering net differences on the feed and permeate sides of a membrane.

Inherent in the definition of permeability, is that a concentration gradient is achieved in conjunction with a pressure gradient. In view of this, the following approach was adopted to determine the value of the term P_{MID} in Eq. (6), which represents the value of the pressure at the PDMS-PEG interface. Using single gas permeability values for CO₂ and N₂ across PDMS (respectively 1.517×10^{-12} and 1.473×10^{-13} mol s⁻¹ m⁻² Pa⁻¹ [12]), the value of the PDMS-PEG interface pressure was determined in the iterative procedure by,

$$P_{MID} = P_{FEED} - \sum_j f_j \cdot D_{PDMS,j} \frac{\Delta C_{PDMS,j}}{\delta_{PDMS} P_{j,PDMS}} \quad (8)$$

Interestingly, the PDMS-PEG interface concentration values showed very little dependence on the value of P_{MID} except at very large values of δ_{PDMS} , since the PDMS layer unit contribution to pressure drop was an order of magnitude less than the PEG membrane material.

In Fig. 5, concentration profiles for CO₂ and N₂ are shown at four different stages of the transient simulation. At $t = 0$, the initial state is shown. The higher diffusivity of the CO₂ is reflected by the establishment of a more linear concentration profile across both the PDMS-PEG layers at 30,000 iterations compared to N₂. Corroboration of the two approaches is shown in the profiles obtained after 10^5 iterations, where the steady state analytical model shows the exact same result as the transient CFD model.

Fig. 6 shows predictions of the Henis model compared to those obtained with the transport based model developed here. For the gas system of CO₂ and N₂ across a PDMS-PEG bi-layer system, at $\delta_{PDMS}/\delta_{PEG}$ values beyond 2, solubility limits produce greater

Fig. 7. Comparison of selectivity predictions from the analytical transport model for the system of O₂ and N₂ across a bi-layer PDMS-PEG system for a range of film thickness ratios at 50 and 10 mol% O₂ feed concentration, against predictions from the Henis model.

flux and selectivity declines than predicted only on the basis of combined permeability resistance. Additionally, the boundary condition applied at the PDMS-PEG interface incorporating local gas mole fractions and solubility, produces a feed concentration dependence for selectivity, observed experimentally, but not predicted by the Henis model. At $\delta_{PDMS}/\delta_{PEG}$ values greater than about 20, the transient CFD approach shows a slightly smaller flux decline than the steady state analytical model. The main difference between the transient and steady state models was the permeate side boundary condition, namely $\nabla(\nabla c) = 0$, which was not forcibly equivalent to setting a steady state molar composition. The transient model allowed an uncoupled determination of the permeate composition.

To further explore the subject, the Henis model was compared to the numerical simulation tested with the mixed gas system of O₂ and N₂, at 50% and 10% O₂ molar feed composition. The purpose of this was to demonstrate a case of a mixed-gas system with components having more similar diffusivity and solubility values (see Table 1) as well as aligned functionality trends relative to the layer materials. In this case, a solubility induced concentration restriction does not arise at the PDMS-PEG interface, and the results of the present transport model essentially match those of the Henis model. Fig. 7 shows the results of these calculations. It can be seen that the predictions of the analytical steady state model do not show any feed concentration dependence in this case. At both the 0.50 and 0.10 mole fraction O₂ feeds, saturation concentrations of O₂ were never required at the PDMS-PEG interface, so that solubility restrictions did not arise and influence the system performance. Thus for the case of bi-layer gas separation of O₂ from N₂, the film resistance model developed by Henis to account for selectivity declines predicts the same results as the basic transport model.

6. Conclusions

As observed experimentally, for the gas separation system of CO₂ and N₂, the transport model developed here predicts accurate and similar selectivity compared to the Henis model at low values of $\delta_{PDMS}/\delta_{PEG}$, the film thickness ratio. As $\delta_{PDMS}/\delta_{PEG}$ increased to values greater than about 5, selectivities lower than those predicted by the Henis model were determined.

The transport based model takes into account solubility based concentration limits at the interface between the two layers, and the solubility based boundary condition there. These solubility related effects have been shown to be responsible for selectivity declines greater than those predicted purely by a film resistance model, and which have been observed experimentally.

Nomenclature

c	concentration [mol m^{-3}]
D	diffusion coefficient [$\text{m}^2 \text{s}^{-1}$]
f_j	mole fraction of gas j
J	diffusional flux [$\text{mol m}^{-2} \text{s}^{-1}$]
P	pressure [Pa]
$P_{j,i}$	permeability of gas j in material i [$\text{mol s}^{-1} \text{m}^{-2} \text{Pa}^{-1}$]
R	ideal gas constant = $8.314 \text{ kg m}^2 \text{s}^{-2} \text{K mol}^{-1}$
S_{ij}	solubility of gas j in material i [$\text{mol m}^{-3} \text{Pa}^{-1}$]
t	time [s]
T	temperature [K]
x	spatial coordinate [m]

Greek letters

δ	layer thickness [m]
ν	partial molar volume [$\text{m}^3 \text{mol}^{-1}$]

Subscripts

mid	position at interface between PDMS and PEG layers
sat	saturation

Abbreviations

PDMS	polydimethyl siloxane
PEI	polyetherimide
PEG	polyethylene glycol
IPN	interpenetrating network
CFD	computational fluid dynamics

The present transport-based approach is able to decouple the effects of diffusivity and solubility, both of which are shown to have independent effects on bi-layer membrane gas separation performance.

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