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Methods to Calculate Gas Diffusion Coefficients of Cellular Plastic Insulation from Experimental Data on Gas Absorption

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INTRODUCTION

THE TECHNOLOGY FOR manufacturing high thermal performance cellular plastic insulations such as polyurethane, polyisocyanurate, phenolics and extruded polystyrene uses gases with low thermal conductivity as captive blowing agents. The gas is captured in the closed cells of the material to achieve higher thermal resistance. But once in contact with the atmosphere, the thermal resistances of these materials gradually decrease. This phenomenon is called aging. The principal reason for aging is known to be the entry of atmospheric gases into the cell structure of the insulation. Atmospheric gases, with higher thermal conductivity than the blowing agents, enter the cells and the blowing agents leave. Usually the molecules of blowing agents are heavier than oxygen and nitrogen molecules in atmosphere. Hence the transport of O_2 and N_2 into the cells is much faster than that of the blowing agent out of the cells. This initially causes a faster decrease in the mole fraction of the blowing agent in the cell gas mixture and therefore a faster decrease in the thermal resistance. Also the rate of aging differs from material to material as illustrated in Figure 1.

For the past two decades, researchers have been developing theoretical models to predict the long term behavior of cellular plastics [1-4]. As Valenzuela and Glicksman stated in a review [5], in order to accurately predict the long term performance of gas filled cellular plastic insulations, it is necessary

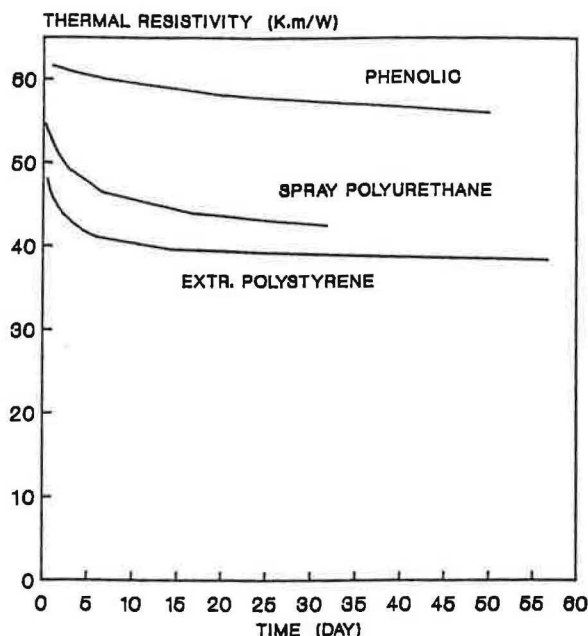


FIGURE 1. Aging of 10 mm thick slices of cellular plastic insulation.

to model the heat transport as well as the mass transport correctly. Improved knowledge of the radiation properties of the polymer matrix has significantly improved the heat transfer models. Mass transfer is invariably modelled as diffusion processes. Once the models are accepted, to use them for numerical calculation, reliable data on a set of gas diffusion coefficients are required. Researchers have developed various techniques to determine these coefficients. Ball et al. [6] calculated the diffusion coefficients of O_2 and N_2 in a polyurethane foam from thermal conductivity data, Norton [1] derived these from gas composition data and Cuddihy and Moacanin [7] calculated the diffusion coefficients from rate of weight loss of samples kept in vacuum. These earlier attempts generated data which differed significantly from method to method [5] and the differences cannot be explained only on the basis of the difference in the test materials.

Recent developments in experimental techniques have resulted in a more consistent set of data. Shankland [8] used a permeance measurement technique. Ostrogosky and Glicksman [9] have reported a steady state method, again based on permeance. Schwartz et al. [10] and Brehn and Glicksman [11] more recently used sorption measurement techniques. The sorption technique has some experimental advantages over the permeance technique,

especially the way in which the specimens are handled. Permeance techniques need a tight separation of two pressure chambers using the test specimens which often results in experimental difficulties. In sorption techniques no such tight sealing is necessary.

To obtain the diffusion coefficient, Schwartz et al. used a finite difference method to analyze their data. Brehn and Glicksman analyzed the data based on an equation proposed by Wilson [12]. The present authors investigated the use of Z -transform [13,14] for the data analyses and compared it with the method used by Brehn and Glicksman and with an improved version of the finite difference method used by Schwartz et al. This paper compares the three methods of analysis and reports a set of data on O_2 and N_2 diffusion coefficients for a polyurethane foam, a polyisocyanurate foam and an extruded polystyrene insulation.

EXPERIMENTAL APPARATUS AND TEST PROCEDURE

The experimental equipment, shown in Figure 2, used to perform the rate of gas diffusion measurements consists of:

1. Test chamber to house the test specimen and to provide test gas pressure conditions
2. Liquid bath to maintain the required temperature of the test chamber and auxiliary chambers
3. Auxiliary equipment such as valves, pressure transducers, etc.
4. Data logger

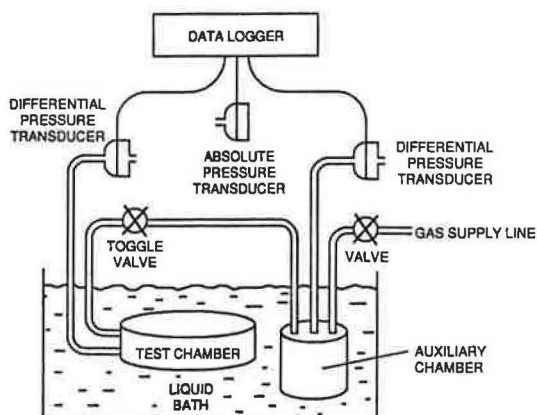


FIGURE 2. Schematic representation of the setup to measure the rate of gas transfer through cellular plastics.

The test chamber can accommodate specimens in the form of discs made out of the foam insulation. These discs were placed in the test chamber and subjected to the initial constant pressure (~ 100 kPa). After the initial settling time to obtain steady state conditions, the chamber pressure was increased by relatively rapid injection of a fixed amount of gas into the chamber from the auxiliary chamber. The chamber gas pressure decreased as the gas diffused into the specimen. The chamber pressure variation with time was monitored continuously for the duration of the experiment.

Further details of the experimental setup and the test procedure are given in Reference [10].

ANALYSIS

The aim of the analysis is to derive a relation between the test chamber pressure variation and time. This relation in turn is used to calculate the gas diffusion and storage characteristics from the experimental data.

The pressure change in the chamber, after a fixed amount of gas is injected into the chamber, is governed by the rate of gas diffusion from the chamber free volume into the specimen. This rate of diffusion (one dimensional) is defined by [15]

$$\frac{\delta^2 C}{\delta x^2} - \frac{1}{\gamma} \cdot \frac{\delta C}{\delta t} = 0 \quad (1)$$

where

C = gas concentration, kg/m^3

x = distance, m

t = time, s

γ = gas diffusion coefficient, m^2/s .

The gas diffusion coefficient, γ , can be expressed as

$$\gamma = \frac{k}{w} \quad (2)$$

where

k = gas permeability, kg/s/m/Pa

w = specific gas storage (which may include gaseous, adsorbed and absorbed states), $\text{kg/m}^3/\text{Pa}$, defined as

$$w = \frac{1}{V} \cdot \frac{dW}{d\phi} \quad (3)$$

where

W = mass of the gas stored in a volume V , kg

V = volume, m^3

ϕ = pressure, Pa

For ideal gas and free volume the specific gas storage is

$$w = 1/(R \cdot T) \quad (4)$$

where

R = gas constant, J/kg/K

T = absolute temperature, K

However, in rigid cellular plastic not all the specimen volume is available for gas storage although apparent storage may increase due to gas absorption by the material. A storage factor, f , can be used to account for this,

$$f = \frac{V_{s,a}}{V_{s,T}} = \frac{w_a}{w_T} \quad (5)$$

where

$V_{s,a}$ = apparent specimen gas storage volume, m^3

$V_{s,T}$ = volume of the specimen, m^3

w_a = apparent cellular plastic specific gas storage, $\text{kg}/\text{m}^3/\text{Pa}$

w_T = free volume specific gas storage, $\text{kg}/\text{m}^3/\text{Pa}$ and from Equations (2) and (4)

$$f = \frac{k \cdot R \cdot T}{\gamma} \quad (6)$$

For convenience, the gas concentration, C , can be expressed in terms of gas pressure

$$C = \phi/(R \cdot T) \quad (7)$$

then under isothermal conditions Equation (1) becomes

$$\frac{\partial^2 \phi}{\partial x^2} - \frac{1}{\gamma} \frac{\partial \phi}{\partial t} = 0 \quad (8)$$

Also, the chamber pressure and the gas mass flow rate into the specimen can be related by (based on the Ideal Gas Law)

$$\frac{d\phi_c}{dt} - \frac{dW_c}{dt} \cdot \frac{R \cdot T}{V_c} = 0 \quad (9)$$

where ϕ_c is chamber pressure, V_c refers to the free chamber volume and W_c gas mass in that volume.

The simultaneous solution of Eqs. (8) and (9) gives the needed relation between chamber pressure variation and gas diffusion characteristics of the specimen.

The calculation procedure can be simplified by using dimensionless time (also called characteristic time) τ [16] where

$$\tau = \gamma \cdot t / L^2 \quad (10)$$

where

L = thickness of the specimen, m

and dimensionless pressure $P_{c,t}$ and $P_{c,\tau}$ (pressure change at anytime expressed as a ratio to initial pressure change) where

$P_{c,t}$ = experimentally determined chamber dimensionless pressure at time t

$P_{c,\tau}$ = calculated chamber dimensionless pressure at dimensionless time τ

The diffusion coefficient could be determined by comparison of the measured (P_t vs. t) and calculated (P_τ vs. τ) chamber pressure decay curves. The "best" match of the two curves would yield the diffusion coefficient and final chamber pressure values for the specimen under consideration.

The determination of the diffusion coefficient, however, can be simplified by the use of a ratio

$$r_{t,f} = (\tau/t) \quad (11)$$

where t is the real time at which the chamber pressure is equal to $P_{c,t}$ (measured values), τ is the dimensionless time such that the calculated $P_{c,\tau}$ and measured $P_{c,t}$ are equal, and f is the storage factor [see Equation (5)] used as a parameter in calculations of $P_{c,\tau}$. Then, from Equations (10) and (11)

$$\gamma_{t,f} = L^2 \cdot r_{t,f} \quad (12)$$

This relation is used to determine the diffusion coefficient and storage factor because if Equation (8) approximates accurately the gas diffusion process in the specimen and if the diffusion coefficient is independent of time and gas concentration then γ must be constant with time. Thus the $\gamma_{t,f}$ curve that is the "best" straight line parallel to the time axis* gives the diffusion coefficient value and the storage factor for the specimen under consideration. This approach to determine the diffusion coefficient is called the **Ratio Method**.

The actual calculations of γ use P_∞ as a parameter, where p_∞ is the chamber pressure at steady state (or is the final chamber pressure), i.e., γ versus t curves are calculated for several assumed P_∞ values. Once the "best" γ and P_∞ values are determined the f factor is calculated as follows: based on Ideal Gas Law

$$P_\infty = \frac{V_c}{V_c + V_{s,a}} \quad (13)$$

then solution of Eq. (5) and Eq. (13) for f gives

$$f = \frac{V_c}{V_{s,r}} \cdot \left(\frac{1}{P_\infty} - 1 \right) \quad (14)$$

To demonstrate this approach to determine γ and f from pressure decay experimental data, a chamber pressure decay was calculated [calculations were based on Equations (8) and (9)] for

$$\gamma = 1.0 \times 10^{-10} \text{ m}^2/\text{s} \text{ and } P_\infty = 0.5$$

This calculated chamber pressure decay was then used as experimental data to calculate γ versus time curves shown in Figure 3.

As expected, γ is constant with time at $p_\infty = 0.5$. For other values of p_∞ the γ versus time curves are not straight lines and lie above and below the $\gamma = 1.0 \times 10^{-10} \text{ m}^2/\text{s}$ value.

Various calculation approaches can be used for the solution of Eqs. (8) and (9). Some methods for various practical reasons, are better suited than the others. For this reason the following three calculation methods were evaluated to assess their suitability:

1. Analytical solution of the gas diffusion equation for a step change of chamber pressure developed by Wilson [12]

* In practice the "best" curve is not exactly a straight line because of the errors in measured data, errors in calculated chamber decay curve and the gas diffusion process may not be exactly linear.

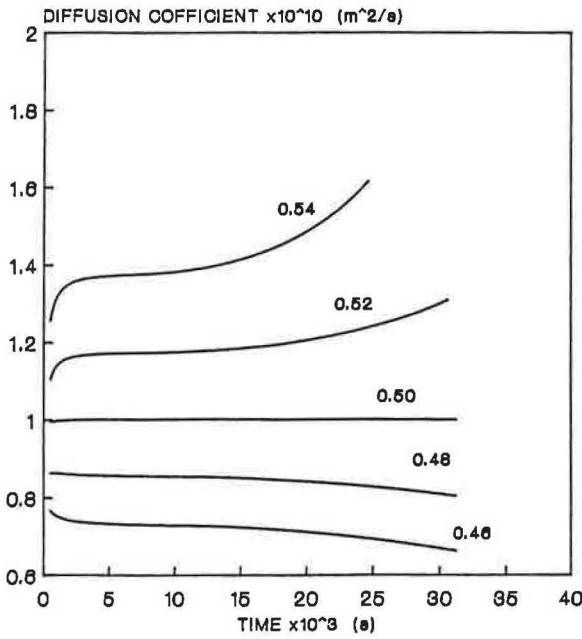


FIGURE 3. Plot of diffusion coefficient curves for a range of chamber final pressures to demonstrate the technique used to determine γ and P_∞ from experimental data; the chamber final pressure values are noted at corresponding curves.

2. *Z*-transfer function method. It is an adaptation of the *z*-transfer function method used in heat transfer problems [13] and solutions of the diffusion equation for ramp excitations [14]
3. Simple explicit finite difference method

ANALYTICAL SOLUTION

The analytical solution (Wilson) for a step change of the chamber pressure at zero time and constant temperature is [12].

$$P_r = \frac{1}{G + 1} + \sum_{n=1} \frac{2 \cdot G}{G \cdot (G + 1) + a_n^2} \cdot \exp(-\alpha_n^2 \cdot \tau) \quad (15)$$

where α_n are the real positive roots of the equation

$$\alpha_n + \tan(\alpha_n) = 0 \quad (16)$$

and

$$G = \frac{1}{P_{\infty}} - 1 \quad (17)$$

The main disadvantages of this method are the lengthy calculations of the roots, α_n ; it is limited to the step chamber pressure change at zero time; uniform initial conditions; and it is applicable only to a linear gas diffusion process. Sample calculated γ curves by this method are shown in Figure 4. This sample calculation used measured chamber pressure decay test data for the polystyrene insulation specimen and N_2 gas maintained at 40°C.

Z-TRANSFER FUNCTION METHOD

The Z -transfer function method, ZTF , is based on two transfer functions

$$K(Z)_{x=0} = \left[\frac{\Delta p}{\Delta x} (z)/p(Z) \right]_{x=0} = \frac{a_0 + a_1 \cdot Z^{-1} + a_2 \cdot Z^{-2} + \dots}{1 + b_1 \cdot Z^{-1} + b_2 \cdot Z^{-2} + \dots} \quad (18)$$

$$K(Z)_{x=L} = \left[\frac{\Delta p}{\Delta x} (z)/p(Z) \right]_{x=L} = \frac{c_0 + c_1 \cdot Z^{-1} + c_2 \cdot Z^{-2} + \dots}{1 + b_1 \cdot Z^{-1} + b_2 \cdot Z^{-2} + \dots} \quad (19)$$

that relate specimen surface gas pressure gradients and chamber pressure. Calculations of the a , b and c coefficients are based on the procedures given in References [13,14] using dimensionless time step $\Delta\tau$.

It should be noted that in the dimensionless form one unique set of $K(Z)_{x=0}$ and $K(Z)_{x=L}$ functions define the relation between the surface gradient and the history of the surface pressure for all homogeneous slabs. An "accurate" set a , b and c coefficients was, therefore, calculated using double precision (~ 14 decimal places) and $\Delta\tau = 0.0002$.

The surface gas transfer, ΔM , during $\Delta\tau$ time period is calculated by

$$\Delta M_{\tau} = \left[\left(\frac{dp}{dx} \right)_{x=0} + \left(\frac{dp}{dx} \right)_{x=L} \right] \cdot L^2 \cdot f \cdot \Delta\tau / (T \cdot R) \quad (20)$$

where (dp/dx) is a function of the transfer function $K(Z)$, the history of surface pressure and the history of surface pressure gradient.

The chamber pressure is calculated by

$$P_{\tau} + \Delta P_{\tau} = (M_{\tau} + \Delta M_{\tau}) \cdot R \cdot T/V_c \quad (21)$$

where M_{τ} is mass of gas in the chamber at time τ .

L=5.098 mm, T=40 C, Gas=N2, XPS, Wilson

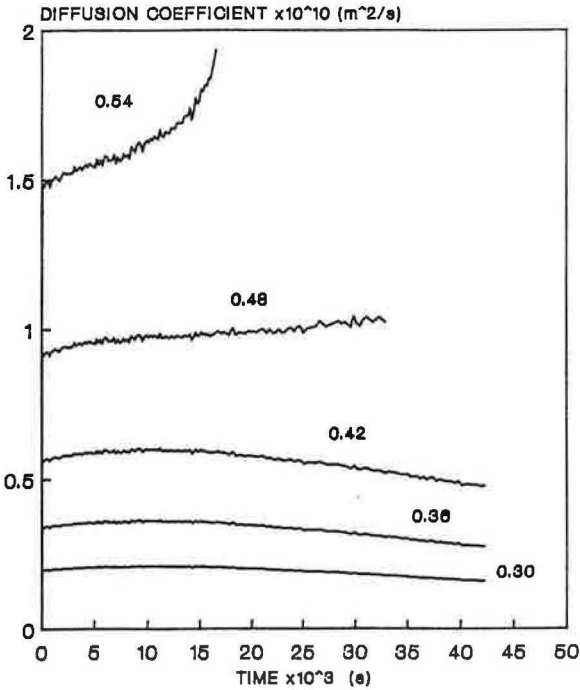


FIGURE 4. Plot of diffusion coefficient curves that were calculated according to Wilson; the chamber final pressures are noted at corresponding curves.

The Z -transfer function method can account for boundary conditions that can be approximated by a straight line segment over the $\Delta\tau$ time period. However, it is still limited in application to the linear diffusion processes. Sample calculated γ curves by ZTF method are shown in Figure 5. This sample calculation used the same test data as it used in calculation of Figure 4 curves.

FINITE DIFFERENCE METHOD

The finite difference method is not as elegant as analytic or z -transfer methods. However, it is simple and it can be applied to account for changes in γ with time, temperature, position and pressure while the other two methods are limited to linear problems.

For the application of the finite difference method, the slab of rigid cellular

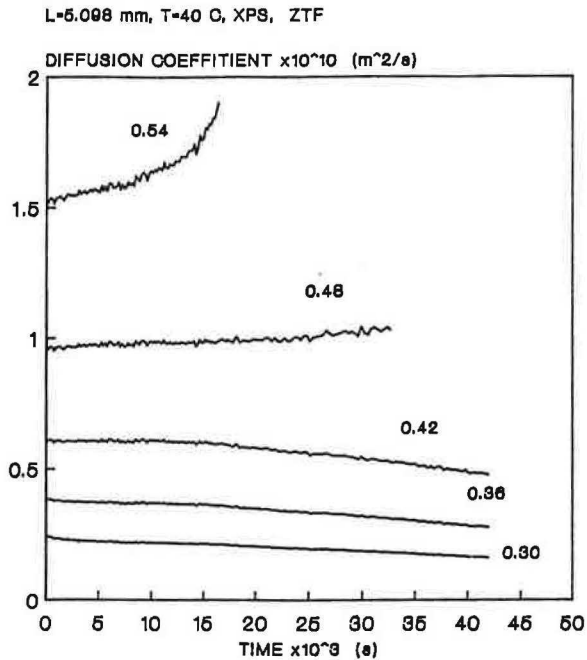


FIGURE 5. Plot of diffusion coefficient curves that were calculated by z-transfer function method; the chamber final pressures are noted at corresponding curves.

plastic is divided (mathematically) into N layers and for each interface the pressures are related (based on the gas mass balance) as follows [17]

$$p_{i-1,\tau} + p_{i+1,\tau} - (2 - U) \cdot p_{i,\tau} = U \cdot p_{i,\tau-\Delta\tau} \quad (22)$$

where

i = interface number

$$U = 1/(\Delta\tau \cdot N^2)$$

A set of equations for all interfaces is denoted by matrix equation

$$|B| \cdot |P_\tau| = |D_\tau| \quad (23)$$

and the solution of this equation gives the gas pressure as

$$|P_\tau| = |B|^{-1} \cdot |D_\tau| \quad (24)$$

Once the pressures for time τ are calculated by the above equation the chamber pressure for time $\tau + \Delta\tau$ are calculated by Eqs. 20 and 21.

The accuracy of finite difference calculations depends on N . Calculations of γ were, therefore, performed using the test data for polystyrene specimen ($L = 5.098$ mm, $T = 40^\circ\text{C}$, Gas - N_2), $N = 20, 30, 40, 50, 60$, and $\Delta\tau = 0.0002$ to demonstrate the influence of N value on the calculated diffusion coefficient, γ . These results indicate that $N = 40$ and $\Delta\tau = 0.0002$ give acceptable accuracy in calculation of γ (see Figure 6).

A sample of diffusion coefficient curves calculated by finite difference method for the same test data that was used in calculations of Figure 4 and Figure 5 curves, are shown in Figure 7. The comparison of curves in Figures 4, 5 and 7 indicates that all three methods produce practically the same diffusion coefficient curves for the same range of assumed chamber final pressures. The criteria, therefore, for selection of the method should be the ease of implementation and the limitations in application of the method.

RESULTS

Pressure decay experiments were performed on three materials using oxygen and nitrogen gases and at several temperatures. The analysis of the ex-

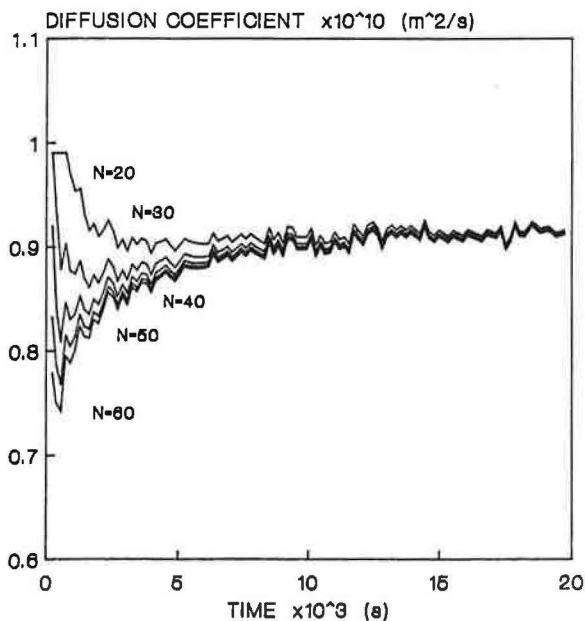


FIGURE 6. Plot of diffusion coefficient curves for a range of N values.

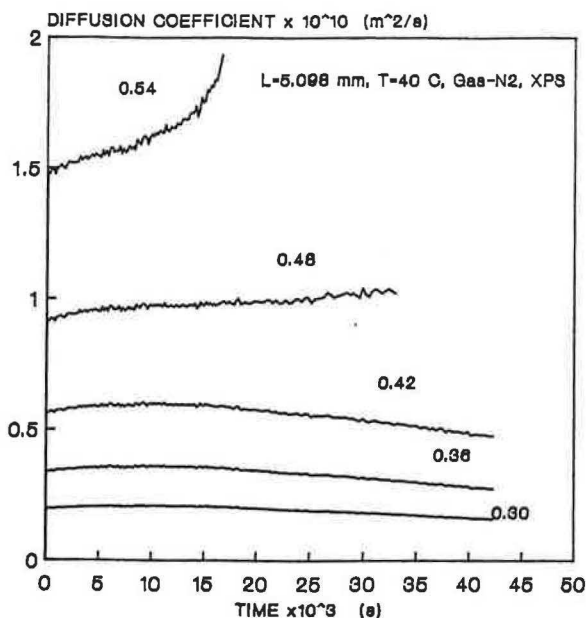


FIGURE 7. Plot of diffusion coefficient curves that were calculated using finite difference method; chamber final pressures are noted at corresponding curves.

perimental data were done using the finite difference calculation approach. Materials, test conditions, the diffusion coefficients, γ , and factor f , determined by the analysis of the experimental data are listed in Table 1.

To demonstrate the technique used as well as the precision of this method, a sample graph for the specimen of extruded polystyrene insulation at 40°C and a range of assumed chamber final pressures is shown in Figure 8.

The results indicate that:

1. Diffusion coefficient, as shown by earlier workers [9], is a strong function of the temperature.
2. The storage factor f in most cases being close to or lower than 1.0 indicates that for the materials investigated here the ability of the polymer matrix to adsorb and absorb O_2 and N_2 is negligible.

As can be seen in Figure 8, the pressure decay experiments can be terminated before the chamber pressure reaches its final steady state value since the γ and P_∞ can be estimated from the data of a partly completed test. Although the accuracy of the estimated γ and f factors increases as more data is used, the accuracy of the measured data decreases because it is difficult to maintain constant test conditions over long periods of time, e.g., N_2 gas test

on some materials may take several hundred hours to approach the new steady state condition. So a compromise must be reached between the cost of the test equipment (assuming that "better" test equipment costs more), the duration of the test (shorter duration permits more tests to be performed with the same equipment) and needed accuracy of γ and f factors.

CONCLUSION

The finite difference approach used to determine diffusion coefficients and storage factors is fast, flexible and easy to apply.

The Ratio Method can be applied to a partly completed test data. This reduces the time needed to determine the diffusion coefficient for relatively low permeability materials and high molecular weight gas.

ACKNOWLEDGEMENT

The authors are indebted to N. Normandin and R. G. Marchand for technical assistance during this investigation.

Table 1. Diffusion coefficients and storage factors for polyurethane (PUR), isocyanurate (PIR) and extruded polystyrene (XPS). All test specimens were prepared from the core of respective samples.

Material	Gas	Temp. °C	$\gamma \cdot 10^{-10}$ m ² /s	f
PUR Density = 31.2 kg·m ⁻³	N ₂	25	.025	0.77
		32	.040	0.73
		40	.060	0.79
	O ₂	25	.22	0.85
		30	.28	0.88
		40	.35	0.96
PIR Density = 30.5 kg·m ⁻³	N ₂	25	.028	1.00
	O ₂	25	.23	1.00
XPS Density = 31.4 kg·m ⁻³	N ₂	25	.58	1.00
		32	.75	0.91
		40	.90	0.95
	O ₂	25	3.8	0.86
		32	4.2	0.89
		40	5.0	0.90

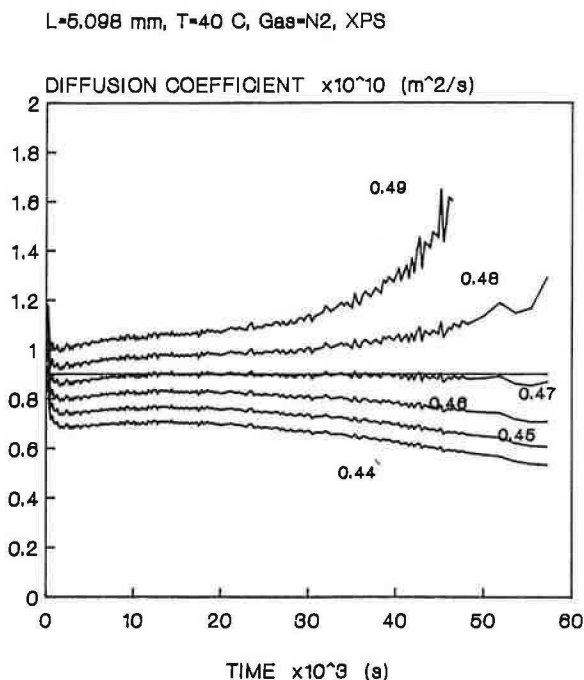


FIGURE 8. Sample graph to demonstrate the technique used to determine diffusion coefficient and chamber final pressure. In this case the estimated diffusion coefficient is $0.90 \times 10^{-10} \text{ m}^2/\text{s}$ and the final pressure is 0.47 shown as a straight horizontal line passing through 0.9×10^{-10} diffusion coefficient value.

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