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TUBES FOR MODIFIED ATMOSPHERE PACKAGING OF FRESH FRUITS AND VEGETABLES: EFFECTIVE PERMEABILITY MEASUREMENT

F. M. Silva, K. V. Chau, J. K. Brecht, S. A. Sargent

ABSTRACT. *Tubes can serve as mediators of gas exchange between the atmosphere inside an impermeable modified package and the surrounding atmosphere. Gas permeabilities through different size tubes (diameter: 0.0065, 0.010, 0.012 and 0.016 m; length: 0.005, 0.010, 0.015, 0.020, 0.022, 0.030, 0.040 m) were measured at 1.5°C, 7°C, and 19°C. It was found that gas permeabilities through tubes were not significantly affected by temperature in the range tested; however tube diameter and length did affect gas permeability. An increase in tube diameter and/or decrease in tube length caused an increase in gas permeability. A regression model relating permeability to tube diameter and length was developed. Oxygen permeability was always greater than carbon dioxide permeability for the same tube dimensions, and permeability ratios (K^*_B/K^*_A) of tubes were found to be between 0.72 and 0.98.*

Keywords. *Modified atmosphere packaging, Tubes, Perforation, Gas permeability, Carbon dioxide, Oxygen.*

The consumer market for fresh, high quality produce is projected to be one of the fastest growing segments of the international food industry during the next five to ten years. Increased consumer demand for greater fresh fruit and vegetable variety, with higher quality, will generate the momentum for this growing market (Malcolm et al., 1993).

After harvest, fresh fruits and vegetables continue their respiration process. To maintain quality, the respiration rate has to be reduced, especially when the products are stored for an extended period or shipped to distant markets. The best way to preserve quality and extend shelf life is by cooling (Ryall and Lipton, 1979; De Wel et al., 1982; Mitchell, 1992). Another method used to reduce respiration and extend shelf life is the modification of the atmosphere surrounding the product. Most products tend to keep longer in atmospheres that are high in carbon dioxide and low in oxygen. Atmosphere modification is usually used as a complement to cooling. Investigations on the use of modified/controlled atmospheres to better maintain the quality of various fresh commodities have shown that the optimum levels of O₂ and CO₂ vary with the commodities (Kader et al., 1989; Kader, 1993; Saltveit, 1993).

A technique that has been developed to provide optimal gas conditions within the product package is modified atmosphere packaging (MAP; Kader et al., 1989; Calderon and Barkai-Golan, 1990). In MAP, the natural process of

respiration of products is used to reduce O₂ and increase CO₂ levels within a package under restricted gas exchange through a barrier (Geeson, 1988). Fruits and vegetables are enclosed in a sealed pack, typically covered with a thin, gas-permeable plastic. The equilibrium levels of O₂ and CO₂ achieved inside the package are functions of the amount of product, its respiration rate, the package gas permeability and area, and the temperature (Chinnan, 1989). With proper temperature control and atmosphere modification, the shelf life of many agricultural products may be extended for long periods.

Several factors influence the design of a suitable MA package: commodity, temperature, optimum atmosphere (% O₂, % CO₂), respiration rate, product weight, and atmosphere outside the package. Based on this information, a packaging film as well as the area of the film have to be selected in such a way that the optimum atmosphere is obtained inside the package.

Modified atmosphere packaging may be accomplished with polymeric films, rigid plastic trays or preformed pouches closed by heat sealing (Geeson et al., 1985; Barmore, 1987; Geeson, 1988). With polymeric films, the desired interior atmosphere is obtained by selecting a film area and permeability that are appropriate for the respiration rate of the product inside the package (Stannett, 1968; Doyon et al., 1991). When impermeable plastics are used, proper gas exchanges may be achieved by using perforations (Watkins et al., 1988; Geeson et al., 1988; Emond et al., 1991; Renault et al., 1994a,b). Because their CO₂/O₂ permeability ratios (PR) are close to one (Emond et al., 1991), perforations or tubes are ideal for products requiring high CO₂ (10-20%) with low levels of O₂ (2-10%) (Emond and Chau, 1990). There are several products with potential applications for MA packaging using perforations: avocado, blackberry, blueberry, cherry, fig, lemon, lime, strawberry, raspberry, spinach, sweet corn, broccoli, asparagus, mushroom, rose, carnation (Emond and Chau, 1990; Emond et al., 1993). In this work, short tubes inserted in the perforations were proposed as gas mediators for a MA package and their effective

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permeability to gases (O_2 and CO_2) was investigated. Air diffusion channels have been used by Ratti et al. (1995) to study modified atmosphere packaging of fresh cauliflower and by Baugerod (1980) in controlled atmosphere storage rooms. However, no measurements were made to determine the effects of diameter and length on gas diffusion through these air diffusion channels.

The permeability of perforations or tubes varies with temperature because the diffusion of gases in air varies as a function of $T^{1.5}$, where T is the temperature in K (Holman, 1990). The rate of diffusion of a gas is also inversely proportional to the square root of its molecular mass, according to Graham's law of diffusion (Sienko and Plane, 1966). For example, the diffusion coefficient of CO_2 in air at $25^\circ C$ and 1 atmosphere is $0.164 \text{ cm}^2/\text{s}$ and that of O_2 in air is $0.206 \text{ cm}^2/\text{s}$, resulting in a PR of 0.8 (Holman, 1990).

The specific objective of this work was to measure the permeabilities to O_2 and CO_2 of different size tubes and establish relations between size, temperature and permeabilities of tubes.

MATERIALS AND METHODS

PERMEABILITY THROUGH TUBES

Effective gas permeabilities for O_2 (K^*_A) and CO_2 (K^*_B) were found by the following procedure. A glass jar fitted with a brass tube through its lid was flushed with a certain gas mixture, then gas samples were drawn periodically from the jar for concentration analysis.

The rate of change of gas concentration inside the jar is given by the following equation (Emond, 1992):

$$\frac{\partial C}{\partial t} = \frac{K^*(C_{\text{out}} - C)}{V} \quad (1)$$

where K^* is the effective gas permeability. Integrating equation 1 for O_2 and CO_2 concentrations leads to:

$$C_{A,t} = C_{A \text{ out}} - (C_{A \text{ out}} - C_{A,t=0}) e^{-\left(\frac{K^*_A t}{V}\right)} \quad (2)$$

$$C_{B,t} = C_{B,t=0} e^{-\left(\frac{K^*_B t}{V}\right)} \quad (3)$$

Taking the natural logarithm of both sides of equations 2 and 3 gives:

$$\ln(C_{A \text{ out}} - C_{A,t}) = \ln(C_{A \text{ out}} - C_{A,t=0}) - \frac{K^*_A t}{V} \quad (4)$$

$$\ln(C_{B,t}) = \ln(C_{B,t=0}) - \frac{K^*_B t}{V} \quad (5)$$

The permeabilities K^*_A and K^*_B can be determined from the slope of the lines given by equations 4 and 5. Eleven different sizes of brass tube were used for the gas permeability tests [inside diameter (m) \times length (m)]: 0.0065×0.005 , 0.0065×0.015 , 0.0065×0.022 , $0.010 \times$

0.010 , 0.010×0.020 , 0.010×0.030 , 0.012×0.010 , 0.012×0.020 , 0.012×0.040 , 0.016×0.010 , and 0.016×0.030 . The diameter sizes were chosen from commercially available tubes and from previous experience, we expected that these tube diameters and lengths would cover the range of permeabilities needed for most MAP applications of interest to us. Permeabilities were determined at $1.5^\circ C$, $7^\circ C$, and $19^\circ C$. This range of temperatures is commonly encountered in the postharvest handling of fresh commodities. Jars with capacity of 0.00177 m^3 were used for the 0.0065-m -diameter tube sizes and 0.00378-m^3 jars were used for all other tube sizes. The high permeabilities of the larger size tubes require a larger jar to reduce the rate of change of the gas concentrations inside the jar.

To start the experiments, a gas mixture containing CO_2 ($> 15\%$), O_2 ($< 5\%$), and N_2 was injected into the jars, which had been placed in a cold room with a preset constant temperature. The shelves where the jars were placed were protected with plastic curtains in order to shield the jars from the air movement caused by the fans on the refrigeration unit inside the cold room. Gas concentrations in each jar were determined five times during a period of 5 to 8 h. At each sampling time, two 1 mL samples were withdrawn from each jar through a rubber septum inserted in the lid of the jar. Three replicates of each tube size and temperature combination were made. The natural logarithms of the gas concentrations were plotted against time to obtain the slopes of the lines and the permeabilities to O_2 and CO_2 were calculated from the slopes.

GAS CONCENTRATION ANALYSIS

Gas samples were analyzed for O_2 and CO_2 with a GowMac, Series 580 gas chromatograph with thermal conductivity detector and a Hewlett Packard Model 3390A integrator. The gas chromatograph was equipped with two columns connected in series. One column was $1.98 \text{ m long} \times 3.175 \times 10^{-3} \text{ m diameter}$ packed with 80 to 100 mesh ColumpakTM PQ and the other was $3.35 \text{ m long} \times 4.763 \times 10^{-3} \text{ m diameter}$ packed with 60 to 80 mesh Molecular Sieve 13X. Temperatures of both columns were set at $40^\circ C$ and detector and injector temperatures were set at $90^\circ C$. The detector current was set at 150 mA during the analysis. The carrier gas was helium at a pressure of 275 kPa with an adjusted 30-mL/min flow rate. Gas samples were withdrawn with 1.0-mL BD plastic syringes with 23G1 BD needles, and 0.5 mL of the gas was injected in the gas chromatograph. Calibrations were done prior to any analysis of the gas samples from any experiment, using a certified standard mixture of 7.18% CO_2 and 7.31% O_2 . Two replicates of each sample were measured.

RESULTS AND DISCUSSION

GAS PERMEABILITY THROUGH TUBES

The natural logarithm of O_2 and CO_2 concentration in the jars increased and decreased linearly over time, respectively. The natural logarithm of the gas concentrations versus time were plotted and used to calculate effective gas permeabilities through tubes. Figure 1 shows such a graph for a 0.003785-m^3 glass jar fitted with a 0.010-m diameter tube, 0.020 m long. The effective permeability to O_2 of the tube was calculated by

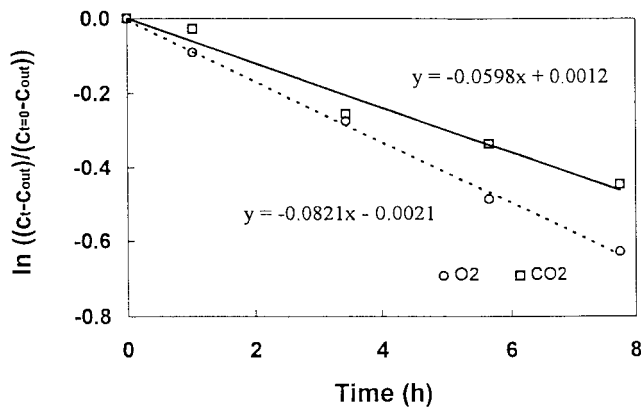


Figure 1—Changes in gas concentration inside the jar fitted with a 10 mm × 20 mm tube at 7°C, replicate 3.

multiplying the slope of the O₂ regression line by the volume of the glass jar (in m³):

$$K^*_A = (0.0821 \text{ 1/h}) \times (1 \text{ h}/3600 \text{ s}) \times 0.003785 \text{ m}^3$$

$$= 8.63 \times 10^{-8} \text{ m}^3/\text{s}\cdot\text{atm}$$

According to theory, which assumes that the permeability coefficient K is a constant, the regression line (shown in fig. 1) should go through the origin of the axes, i.e., there is no y-intercept. However, when a straight line regression was fitted to the data, there were small, positive y-intercepts. This indicates that there was a time delay at the start of the test, and/or the diffusion coefficient was slightly higher at the beginning of the process. We chose not to force the regression line through the origin because we did not want the possible effects of the time delay and/or slightly higher permeation at the beginning of the diffusion process to interfere with the value of the long-term permeability coefficient. In any case, since the y-intercepts were very small, either approach would give very similar results.

The K* for O₂ and CO₂ were calculated from the replicates of each tube size tested at each temperature. Table 1 lists the average of all the effective gas permeability values. In all tubes, permeability to O₂ was always larger than permeability to CO₂ by 2% to 38%, and consequently the permeability ratio (PR = K_B*/K_A*) was always lower than one; PR varied from 0.72 to 0.98. This

result is consistent with Graham’s law of gas diffusion, which states that gas diffusion is inversely proportional to the square root of the molecular weight. For the tube sizes and temperatures tested, the values for permeabilities varied between $1.92 \times 10^{-8} \text{ m}^3/\text{s}\cdot\text{atm}$ (K_A* at 1.5°C with tube dimensions of 0.065 m × 0.022 m) and $44.82 \times 10^{-8} \text{ m}^3/\text{s}\cdot\text{atm}$ (K_A* at 19°C, tube dimensions 0.016 m × 0.010 m).

EFFECT OF TEMPERATURE

There appeared to be a very slight increase in permeability with temperature (fig. 2), but statistical analysis, to be presented later in this article, showed that temperature was not a significant factor. It was expected that the permeability would be proportional to T^{1.5} when T was the temperature in degrees Kelvin. The temperatures used in the experiments were 1.5°C (274.5°K), 7°C (280°K) and 19°C (292°K). It was therefore expected that the permeability would increase by 9.7% when the temperature was changed from 1.5°C to 19°C [(292/274.5)^{1.5} = 1.097]. From experimental data, an increase in temperature from 1.5°C to 19°C results in permeability increase of about 8 to 10% depending on tube size and O₂ or CO₂ gas type. This slight increase was probably within the variability of the experimental data and

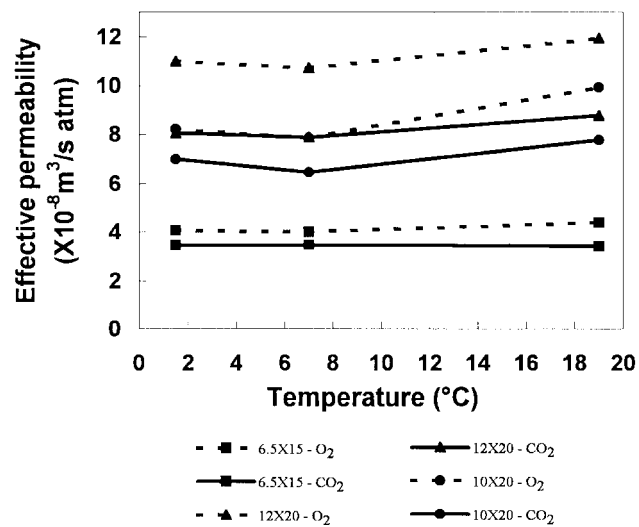


Figure 2—Effect of temperature on tube permeability to O₂ and CO₂.

Table 1. CO₂ and O₂ effective permeabilities of different size tubes at 1.5°C, 7°C, and 19°C

Tube Dimension D × L (m × m)	K* (1.5°C) (× 10 ⁻⁸ m ³ /s·atm)		K* (7°C) (× 10 ⁻⁸ m ³ /s·atm)		K* (19°C) (× 10 ⁻⁸ m ³ /s·atm)	
	CO ₂	O ₂	CO ₂	O ₂	CO ₂	O ₂
0.0065 × 0.005	7.10 ± 0.49	8.08 ± 0.30	6.29 ± 0.05	8.00 ± 0.68	7.43 ± 0.02	9.56 ± 0.61
0.0065 × 0.015	3.45 ± 0.25	4.05 ± 0.27	3.47 ± 0.32	4.01 ± 0.33	3.91 ± 0.27	4.39 ± 0.38
0.0065 × 0.022	1.92 ± 0.76	2.44 ± 0.63	2.72 ± 0.21	2.77 ± 0.34	2.59 ± 0.19	3.26 ± 0.50
0.010 × 0.010	10.94 ± 0.08	13.24 ± 1.73	10.97 ± 0.33	12.62 ± 2.49	11.79 ± 3.98	12.90 ± 4.84
0.010 × 0.020	6.99 ± 1.10	8.20 ± 1.74	6.45 ± 0.12	7.86 ± 0.48	7.76 ± 0.59	9.92 ± 0.74
0.010 × 0.030	4.25 ± 0.96	4.77 ± 0.94	4.56 ± 0.3	6.31 ± 0.66	5.29 ± 0.16	6.48 ± 0.24
0.012 × 0.010	19.39 ± 0.78	24.36 ± 2.04	19.31 ± 3.07	21.96 ± 2.77	15.31 ± 1.11	21.28 ± 0.59
0.012 × 0.020	8.05 ± 0.48	10.99 ± 1.04	7.88 ± 0.39	10.71 ± 0.40	8.75 ± 0.56	11.93 ± 1.21
0.012 × 0.040	4.64 ± 0.09	5.25 ± 0.11	5.20 ± 1.19	5.83 ± 0.39	6.09 ± 1.53	7.15 ± 1.52
0.016 × 0.010	20.86 ± 0.65	25.66 ± 1.2	29.06 ± 4.40	36.37 ± 4.13	35.74 ± 7.25	44.82 ± 8.47
0.016 × 0.030	16.56 ± 0.22	20.85 ± 0.92	12.28 ± 1.24	15.73 ± 1.61	12.52 ± 0.19	17.20 ± 1.97

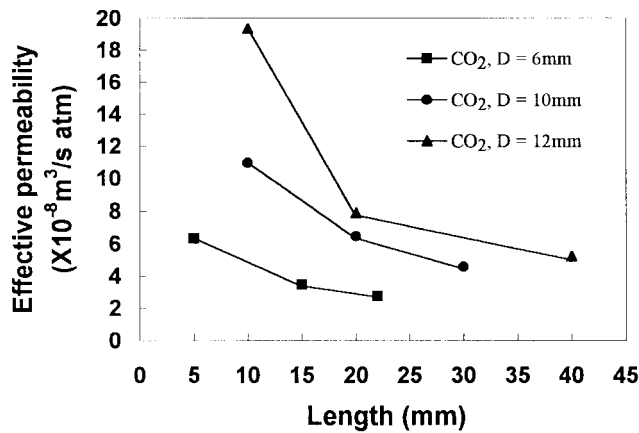


Figure 3—Effect of tube length on CO₂ permeability at 7°C.

this was not considered significant by the statistical analysis.

EFFECT OF TUBE DIMENSIONS

Different tube lengths ranging from 0.005 m to 0.040 m were used in these experiments. An increase in the length of the tubes caused a decrease in permeability (fig. 3). However, the rate of this reduction in permeability decreased with increasing length. The effect of tube length on permeability also decreased with decreasing diameter. Figure 3 shows the effect of length on CO₂ permeabilities of three different tubes at 7°C. The same trends were observed at 1.5°C and 19°C.

Tube diameters tested varied from 0.0065 m to 0.016 m. Larger diameter tubes had much larger gas permeability values for the same temperature and tube length (fig. 4). Gas permeability was much more sensitive to changes in tube diameter than tube length; small changes in diameter had a much larger impact on the permeability than small changes in tube length. For example, in the 0.010-m tube length, when the diameter was increased from 0.010 m to 0.012 m (20% increase) the CO₂ permeability increased from 10.97 × 10⁻⁸ m³/s·atm to 19.31 × 10⁻⁸ m³/s·atm (76% increase). However, an increase in length from 0.010 m to 0.020 m for the 0.010-m diameter tube (100% increase) only resulted in a CO₂ permeability decrease from 10.97 × 10⁻⁸ m³/s·atm to 6.45 × 10⁻⁸ m³/s·atm (60% decrease).

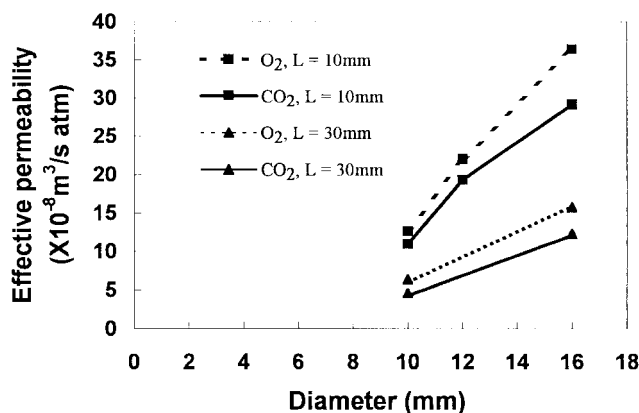


Figure 4—Effect of tube diameter on gas permeabilities at 7°C.

EMPIRICAL EQUATIONS FOR GAS PERMEABILITY PREDICTION

To relate the effects of temperature (T), tube diameter (D), and length (L) on permeability, a multiple regression with forward selection procedure was applied and Statistical Analysis System (SAS) for Linear Models was used for calculations. The procedure was started by fitting simple regression models with individual variables; then, based on those models, more complex regression models were adopted and evaluated. As a variable, temperature was eliminated at the beginning of the procedure because the probabilities for all temperature terms (T and T²) were larger than 0.1. If the probability was below 0.1, the estimated coefficient for that variable was significantly different from zero, after adjusting for the other term in the model. More complex regression models that included higher order terms for the variables L, D and their combinations (interaction) were then attempted. The following model was found to best describe the relationship between the diameter and length and effective gas permeability:

$$K^* = a_1 + a_2(D^2) + a_3(L) + a_4(L^2) + a_5(D^2)(L) \quad (6)$$

In the above equation, effective permeability (K*) is in m³/s·atm, diameter (D) and length (L) in m. By using the SAS for Linear Models statistical package, the coefficients, a_i, that minimized the R² were computed for both O₂ and CO₂ permeability (table 2). The significance of the R² for O₂ and CO₂ are both less than 0.001.

EXAMPLE OF A PERFORATION-GENERATED MODIFIED ATMOSPHERE PACKAGE (MAP) WITH STRAWBERRIES

Equation 6 has been successfully used to design modified atmosphere packages for strawberries under different temperatures and surrounding atmospheres (Silva, 1995). One example from this study is shown here to illustrate how well the system works. In this example, three glass jars each fitted with one of the three following tubes were used: 0.0065 m × 0.0015 m (diameter × length), 0.0065 m × 0.0022 m, and 0.002 m × 0.0040 m. The first two jars were 0.0018 m³ in volume and held 0.5 kg of strawberries. The third jar had a volume of 0.0038 m³ and held 1 kg of strawberries. The procedure outlined by

Table 2. Coefficients for equation 6

Coefficient (a _i)	Values for O ₂ Permeability	Values for CO ₂ Permeability
a ₁	5.4182012 × 10 ⁻⁸	5.0726526 × 10 ⁻⁸
a ₂	1.7316644 × 10 ⁻³	1.393864 × 10 ⁻³
a ₃	-5.749949 × 10 ⁻⁶	-5.225988 × 10 ⁻⁶
a ₄	1.164264 × 10 ⁻⁴	1.132462 × 10 ⁻⁴
a ₅	-3.38719 × 10 ⁻²	-2.91138 × 10 ⁻²
	R ² : 0.89	R ² : 0.88

Table 3. Experimental and predicted results for strawberries at 7°C under controlled atmosphere at 14.8% O₂ and 8.3% CO₂

Tube Size D × L (m × m)	Predicted		Experimental ± S.D.	
	% O ₂ Inside	% CO ₂ Inside	% O ₂ Inside	% CO ₂ Inside
0.0065 × 0.015	11.2	10.9	12.9 ± 0.3	11.1 ± 0.3
0.0065 × 0.022	9.8	11.8	11.8 ± 0.5	12.2 ± 0.4
0.012 × 0.040	10.0	11.8	11.0 ± 0.4	13.3 ± 0.4

Talasila et al. (1995) along with equation 6 above and the strawberry respiration equations given by Talasila (1992) were used to determine the tube sizes, strawberries weights and the expected resulting atmospheres. Table 3 shows the predicted values and the steady-state experimental values, which are the average of eight replicates. There is good agreement with the predicted and experimental values. More details and results of other experiments are given by Silva (1995).

CONCLUSIONS

In the range of temperatures tested (1-19°C), tube permeability to O₂ and CO₂ is not significantly affected by temperature. Gas permeability exhibits large increases with increasing tube diameter and smaller decreases with increasing tube length. O₂ permeabilities are always higher than CO₂ permeabilities for the same tube. Tube permeability ratios vary from 0.72 to 0.98. The applicability of an impermeable package fitted with a tube for MAP was demonstrated for strawberries.

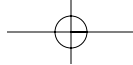
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SYMBOLS

- a₁, a₂,... = coefficients for a regression model
 C = gas partial pressure (atm)
 D = tube diameter (m)
 K* = effective gas permeability through a tube (m³/s-atm)
 L = tube length (m)
 PR = permeability ratio, dimensionless



t = time (s)
T = temperature, K (unless specified otherwise)
V = volume (m³)

SUBSCRIPTS

A = oxygen (O₂)
B = carbon dioxide (CO₂)
out = refers to the outside of the package/jar
t = time (s)

