Modelling O_2 and CO_2 exchange for development of perforation-mediated modified atmosphere packaging

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Abstract

Perforation-mediated modified atmosphere packaging relies on the use of macro perforations or tubes to control the O_2 and CO_2 exchange and create the desired atmosphere inside an otherwise gas-tight package. In this work, the O_2 and CO_2 exchange through a single tube was studied. Different temperatures (5–20°C) and tube dimensions (length from 9 to 17 mm and diameter from 6 to 30 mm) were tested. O_2 and CO_2 mass transfer coefficients were determined according to a lumped mass transfer capacitance model that yielded a good description of the gas transfer. Temperature in the range tested did not show a significant effect on the mass transfer coefficients. A multiplicative non-linear equation was found to yield a good prediction of the dependence of the O_2 mass transfer coefficient on tube diameter and length. The ratio between the CO_2 and O_2 mass transfer coefficients, an important parameter in the design of MAP, was 0.81 and none of the factors tested influenced its value.

Nomenclature

D	tube diameter (m)
K	mass transfer coefficient ($m^3\ s^{-1}$)
K^{pred}	mass transfer coefficient predicted by the model ($m^3\ s^{-1}$)
L	tube length (m)
T	temperature (°C)
t	time (s or min)
V	package volume (m^3)
y	volumetric concentration (%)
y^e	outside volumetric concentration (%)
y^{exp}	experimental volumetric concentration (%)
y^i	initial volumetric concentration (%)
y^{pred}	volumetric concentration predicted by the model (%)

1. Introduction

Fruits and vegetables are highly perishable products that continue their metabolic processes after harvest. The use of modified atmosphere packaging (MAP) in postharvest preservation of these horticultural commodities is one important technique to reduce losses and maintain quality throughout the distribution system (Anzueto & Rizvi, 1985; Zagory & Kader, 1988; Cameron, 1989; Geeson, 1989; Nakhasi, Schlimme & Solomos, 1991). MAP is an atmosphere modification that relies on the interplay between the natural process of produce respiration and gas exchange through the package. Due to respiration, there is a build up of carbon dioxide (CO_2) and a depletion of oxygen (O_2) that is regulated by the gas exchange through the package so that, at equilibrium, adequate O_2 and CO_2 concentrations are reached. The low levels of O_2 and the high levels of CO_2 in MAP can potentially reduce respiration rate, ethylene sensitivity and production, decay and physiological changes, namely oxidation, with the resultant benefit of extending the storage life of the fresh produce (Kader, Zagory & Kerbel, 1989; Saltveit, 1993).

Although flexible polymeric films are widely used in MAP, these materials have some limitations because of

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their structure and permeation characteristics: (i) films are not strong enough for large packages, (ii) film permeability characteristics change unpredictably when films are stretched or punctured, (iii) some films are relatively good barriers to water vapour, causing condensation inside packages when temperature fluctuations occur and consequently increasing susceptibility to microbial growth, (iv) film permeability may be affected by water condensation, (v) the uniformity of permeation characteristics of films is not yet satisfactory, (vi) film permeability is too low for high respiring products, and (vii) products that require high CO_2/O_2 concentrations may be exposed to anaerobiosis because of the high ratio of CO_2 to O_2 permeability coefficients (Cameron, 1989; Kader et al., 1989; Emond & Chau, 1990; Vries-Paterson, Jones & Cameron, 1991; Beit-Halachmy & Mannheim, 1992; Exama, Arul, Lencki, Lee & Toupin, 1993; Joles, Cameron, Shirazi, Petracek & Beaudry, 1994; Cameron, Talasila & Joles, 1995).

The limitations of polymeric films and the growing interest in MAP for (i) bulk packages, (ii) high respiring products, and/or (iii) fresh-cut products is leading to the development of new packaging materials and systems. Emond and Chau (1990) presented the concept of perforation-mediated MAP as an alternative system. Perforation-mediated MAP relies on the use of macro perforations in an otherwise gas tight-container (e.g., a plastic material or a corrugated cardboard with an impermeable liner) to control O_2 and CO_2 exchange through the package. Perforations may be drilled through the package or alternatively tubes with selected dimensions may be inserted through the package (for the sake of simplicity, the word perforation will be used throughout this paper, both to designate tubes or perforations). This system may therefore be applied in bulk packages and, because of the small β ratio, the ratio between CO_2 to O_2 mass transfer coefficients (Mannapperuma, Zagory, Singh & Kader, 1989; Emond, Castaigne, Toupin & Desilets, 1991), it is suitable for commodities requiring high CO_2 concentrations with relatively high O_2 concentrations (e.g., strawberries, blueberries, raspberries, blackberries). Non-uniformity of concentrations inside the package and water loss of product near the perforations may be pointed out as limitations of this type of packaging (Fonseca, Oliveira, Chau & Brecht, 1997). Emond et al. (1991) simulated the use of this packaging system with strawberries and Emond (1992) studied its application in bulk packages of blueberries. Silva (1995) developed a shipping/retail system for mixed loads using a perforation-mediated MAP for strawberries.

The evolution of gas composition (O_2 or CO_2) due to the gas exchange through a single perforation has been described by a lumped mass capacitance model (Emond et al., 1991), when there is no produce in the package,

$$y = y^{e} + (y^{i} - y^{e}) \exp\left(-\frac{K}{V}t\right), \quad (1)$$

where y is the volumetric concentration at time t within the package, y^{e} the volumetric concentration in the surrounding atmosphere, y^{i} the initial volumetric concentration within the package, V the package volume and K the mass transfer coefficient, which includes the effect of the cross-sectional area and length of the perforation.

The gas exchange rate is controlled by the number and dimensions of the perforations and may be expected to depend on temperature and also on atmospheric pressure. The development of perforation-mediated MAP thus depends on the knowledge of the effect of these variables on the mass transfer coefficients. Emond et al. (1991) studied the O_2 and CO_2 exchange through a perforation drilled in an impermeable plexiglass box and Silva (1995) studied the exchange through a tube inserted in the lid of a glass jar. Emond et al. (1991) and Silva (1995) developed empirical and additive models, with eight and five parameters, respectively, relating O_2 and CO_2 mass transfer coefficients with temperature, diameter and length of the perforation. These models were, however, both based on a small number of experimental runs and the effect of the deviation between measured and predicted mass transfer coefficients on the prediction of gas concentration changes was not evaluated. Indeed, Emond et al. (1991) concluded that further studies were needed to fully assess the effects of perforation dimensions.

The general objective of this work was thus to conduct a systematic study of the gas exchange through a perforation in a gas-tight package, with the aim of developing a simple yet accurate mathematical model to relate O_2 and CO_2 mass transfer coefficients to temperature and perforation dimensions.

2. Materials and methods

2.1. Experimental procedure

Brass tubes with different dimensions (Table 1) were inserted half inside half outside across tinplate twist-off lids fitted with stoppers for gas sampling and semi-rigid tubes to feed a gas mixture. Glass jars (2 dm^3) sealed with these lids (gas-tight package simulation) were put in a walk-in controlled temperature cold room. The temperature of the room was maintained within 1°C of the set temperature (5–20°C). The jars were flushed with 20–25% CO_2 and the balance nitrogen. The changes in gas concentration with time were determined by taking 100 μl gas samples from time to time with 1 ml TLL Hamilton (Reno, Nevada, USA) glass syringes fitted with KF Hamilton needles. In order to assess the

Table 1
Factors and levels used in the experiment for model building (in parenthesis: average ± standard deviation)

Factors	Levels			
Temperature (°C)	5	10	15	20
Diameter (mm)	9 (8.68 ± 0.05)	11 (11.0 ± 0.1)	14 (14.1 ± 0.2)	17 (17.0 ± 0.1)
Length (mm)	6 (6.48 ± 0.29)	10 (11.7 ± 1.0)	20 (21.0 ± 1.3)	30 (30.6 ± 1.0)

assumption of jar seal effectiveness, a gas mixture of known composition was flushed into a jar closed with a lid without perforations, the jar sealed, and the gas composition measured over time; no variations of concentration were noticed.

2.2. Gas concentration analysis

The gas samples were analysed with a 5890 series II HP (Boeblingen, Germany) gas chromatograph with a thermal conductivity detector and a HP 3396 series II integrator. The gas chromatograph used a HayeSep CRS (Addison, Illinois, USA) packed column. The gas carrier was helium at 390 kPa. Temperatures of the column, injector and detector were set at 25°C, 100°C and 140°C, respectively. The gas samples were injected in the gas chromatograph. Sampling presumably had no influence on jar concentrations, as the jar volume was much greater than total sampling volume. Calibration was performed using a calibration mixture of 6.99% O_2/6.99% CO_2/86.02% N_2.

The composition of the surrounding atmosphere was measured experimentally (eight replicates taken randomly) and found to be equal to $y^e_{O_2} = 20.358 \pm 1.197\%$ and $y^e_{CO_2} = 0.793 \pm 0.680\%$.

2.3. The experimental design

A set of two replicate experimental runs was performed with all combinations of four levels of the three factors under study (temperature, tube length and tube diameter), in a total of 128 treatments (Table 1). The tube dimensions selected were similar to those used in published works (Emond et al., 1991; Silva, 1995) and the range of temperature covers normal transport and distribution conditions.

An additional set of eight experimental runs was conducted with all the combinations of the extreme levels of temperature, tube diameter and tube length, in order to validate the mathematical model developed on the basis of the first experiment.

2.4. Parameter estimation and model building

Mass transfer coefficients for O_2 and CO_2 were estimated by fitting Eq. (1) to the experimental data by non-linear regression using the Statistica software (release 5.1, 97 edition, Statsoft, Tulsa, Oklahoma, USA). Model building to determine an equation relating mass transfer coefficients to tube dimensions was also developed using the Statistica software.

3. Results and discussion

3.1. The mass transfer coefficients

Fig. 1 shows typical results of the fit of the mathematical model described by Eq. (1) to the experimental data. The scatter plots of the experimental data (data not shown) showed that the model adequately describes O_2 and CO_2 exchange through tubes. The sample coefficient of determination (R^2) varied between 89.2% and 100.0%. Table 2 shows the estimates of the mass transfer coefficients, together with the β ratios, and relevant statistical data, for the minimum and maximum levels of temperature. Estimates varied from 4.74×10^{-8} to 43.2×10^{-8} $m^3\ s^{-1}$ for O_2 and from 3.55×10^{-8} to 39.4×10^{-8} $m^3\ s^{-1}$ for CO_2, being of the same order of magnitude as published results (Emond et al., 1991; Emond, 1992; Silva, 1995). If one considers the process to take place by Fickian diffusion, these values would correspond to diffusivity values from 0.77×10^{-5} to 3.5×10^{-5} $m^2\ s^{-1}$ for O_2 and from 0.79×10^{-5} to 3.1×10^{-5} $m^2\ s^{-1}$ for CO_2. These values are of the same order of magnitude as those measured for diffusion in air (Holsen & Strunk, 1964; Loncin & Merson, 1979; Mannapperuma et al., 1989), as might be expected because of the large size of the tubes.

An analysis of variance led to the conclusion that temperature had no significant effect ($P > 0.05$) on the mass transfer coefficients. Similar results were obtained by Silva (1995), whereas Emond et al. (1991) reported a positive effect, even greater than that expected for diffusion in air. Our results may be due to different boundary conditions at the perforation outlet: at the higher temperatures tested, the temperature control of the cold room almost did not operate, because this temperature was very close to the ambient outside room temperature, while at the lower temperatures, the refrigeration system operated for longer periods, improving the air circulation within the cold room; thus, the expected positive effect of temperature could have been masked by the different conditions of air

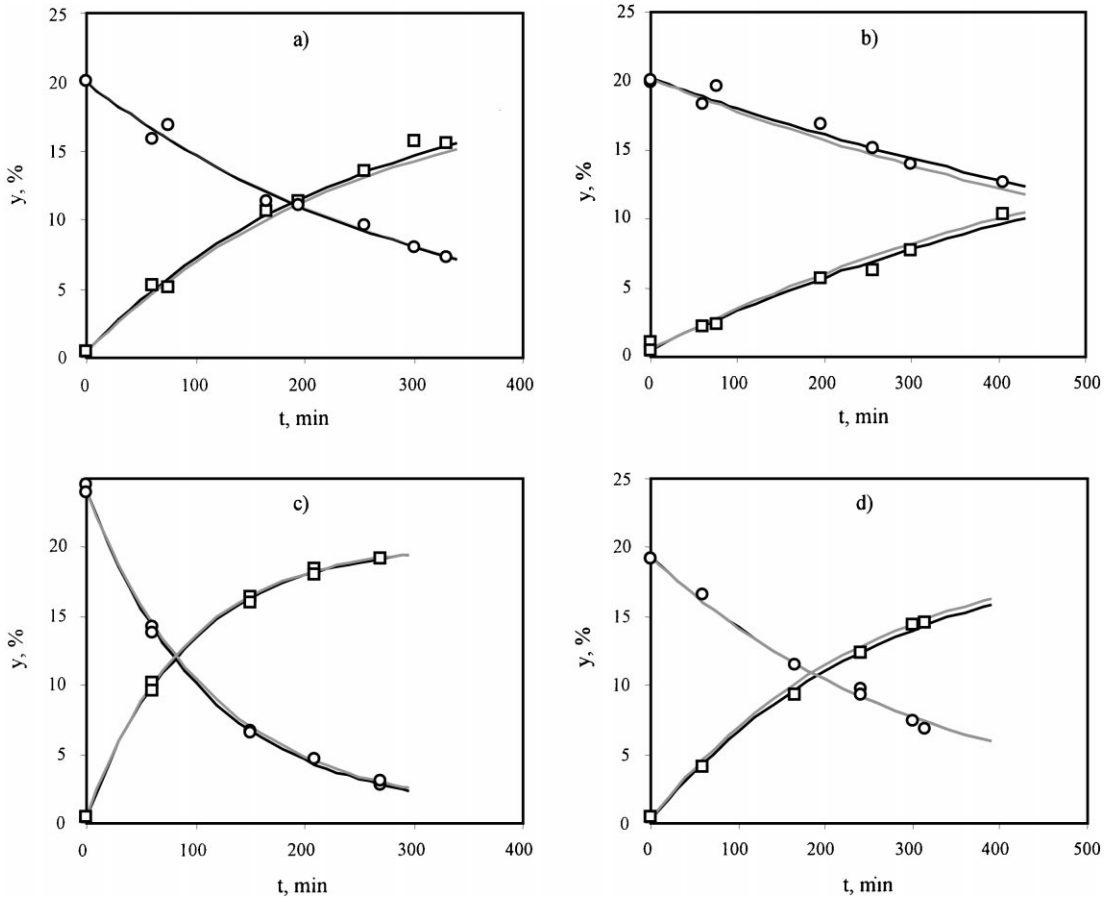

Fig. 1. Typical examples of the changes of O_2 and CO_2 concentrations over time: (a) $D = 8.7$ mm, $L = 6.7$ mm, $T = 15°C$; (b) $D = 8.7$ mm, $L = 29.5$ mm, $T = 15°C$; (c) $D = 17.1$ mm, $L = 6.4$ mm, $T = 15°C$; (d) $D = 16.9$ mm, $L = 31.8$ mm, $T = 10°C$, (□ O_2, ○ CO_2, — individual model, ═══ overall model).

circulation in the cold room for different temperatures. Nevertheless, one should also take into consideration that the range of temperatures tested was very small and thus the temperature effects were quite reduced: the diffusion coefficient reported by Mannapperuma et al. (1989) for O_2 at 5°C is 1.83×10^{-5} and at 20°C is 1.98×10^{-5} m^2 s^{-1}, an increase smaller than 10%, whereas for the same variation of temperature the permeability coefficient of O_2 in PVC was reported to increase over 200%.

The effect of the tube length on the mass transfer coefficients was negative, although smaller than that predicted by Fick's law, probably because of end effects: increasing tube length from 6 to 30 mm decreased the mass transfer coefficient by approximately 3.3-fold, whereas Fickian diffusion would predict a decrease of 5-fold. Tubes with larger diameters showed faster mass transfer rates, but again the effect of increasing the diameter was smaller than what would be expected from Fick's law: increasing the diameter from 9 to 17 mm increased the mass transfer coefficient by 2.8-fold, in comparison to an increase of 3.6-fold predicted by Fick's law.

The β ratio varied between 0.655 and 1.135, averaging 0.813, but no relationship was found between this parameter and the tube dimensions, as expected, because the experimental conditions may similarly affect both the O_2 and CO_2 coefficients.

3.2. Model building

Applying logarithms to the dimensionless concentrations of CO_2 and O_2, based on Eq. (1), and dividing both equations, we obtain:

$$y_{CO_2} = y^{e}_{CO_2} + \left(y^{i}_{CO_2} - y^{e}_{CO_2}\right) \exp\left[\beta \ln\left(\frac{y_{O_2} - y^{e}_{O_2}}{y^{i}_{O_2} - y^{e}_{O_2}}\right)\right]. \quad (2)$$

This equation allows for the estimation of β, without requiring the calculation of the mass transfer coefficients and thus improving the estimation of this parameter. Non-linear regression of Eq. (2) to the whole data set led to $\beta = 0.81 \pm 0.01$ ($R^2 = 97.9\%$). This value is lower than the value reported by Emond et al. (1991) but

Table 2
O_2 and CO_2 mass transfer coefficients estimated by application of Eq. (1) to the experimental data, β ratios and relevant statistical data

D (mm)	L (mm)	T (°C)	$(KO_2 \pm SD) \times 10^8$ (m^3/s)	R^2 (%)	$(KCO_2 \pm SD) \times 10^8$ (m^3/s)	R^2 (%)	β ratio ± SD
8.7	6.7	5	16.14 ± 0.48	99.5	13.46 ± 0.65	98.5	0.834 ± 0.068
8.7	6.7	20	15.67 ± 0.82	99.3	11.62 ± 0.61	99.1	0.742 ± 0.100
8.7	12.4	5	10.56 ± 0.51	98.5	8.53 ± 0.37	98.5	0.808 ± 0.080
8.7	12.4	20	9.41 ± 0.51	98.3	7.28 ± 0.61	95.8	0.773 ± 0.129
8.6	19.8	5	9.93 ± 0.37	99.3	9.02 ± 0.37	99.1	0.908 ± 0.061
8.6	19.8	20	10.16 ± 0.30	99.4	7.29 ± 0.41	98.2	0.718 ± 0.088
8.7	29.5	5	4.84 ± 0.14	99.5	3.69 ± 0.30	95.3	0.762 ± 0.114
8.7	29.5	20	4.74 ± 0.20	99.2	3.56 ± 0.60	89.2	0.752 ± 0.232
11.0	6.7	5	16.10 ± 0.98	98.3	11.93 ± 0.68	97.9	0.741 ± 0.113
11.0	6.7	20	16.31 ± 0.34	99.7	13.83 ± 0.47	99.1	0.848 ± 0.047
11.2	11.6	5	13.29 ± 0.41	99.4	11.23 ± 0.51	98.6	0.845 ± 0.065
11.2	11.6	20	13.46 ± 0.27	99.7	10.21 ± 0.30	99.3	0.759 ± 0.047
10.9	21.3	5	9.10 ± 0.31	99.2	6.69 ± 0.31	98.4	0.735 ± 0.077
10.9	21.3	20	8.66 ± 0.37	98.6	7.00 ± 0.44	96.4	0.808 ± 0.095
11.0	31.1	5	7.16 ± 0.34	98.3	4.88 ± 0.24	98.1	0.682 ± 0.099
11.0	31.1	20	6.61 ± 0.11	99.9	5.29 ± 0.20	98.7	0.800 ± 0.053
13.9	6.1	5	26.05 ± 0.81	99.6	20.88 ± 0.61	99.6	0.802 ± 0.053
13.9	6.1	20	27.36 ± 1.86	98.6	24.09 ± 1.08	99.1	0.880 ± 0.092
14.0	10.3	5	22.74 ± 0.78	99.5	17.99 ± 0.64	99.2	0.791 ± 0.063
14.0	10.3	20	21.96 ± 0.98	99.1	17.89 ± 0.47	99.6	0.815 ± 0.064
14.3	22.7	5	13.95 ± 0.85	97.5	9.13 ± 0.44	98.2	0.655 ± 0.119
14.3	22.7	20	13.61 ± 0.85	97.7	11.37 ± 0.37	99.2	0.835 ± 0.084
14.2	30.0	5	11.06 ± 0.88	95.4	8.50 ± 0.64	95.9	0.768 ± 0.142
14.2	30.0	20	10.83 ± 0.61	98.0	7.76 ± 0.44	98.0	0.717 ± 0.111
17.1	6.4	5	39.61 ± 1.91	99.6	32.64 ± 1.60	99.6	0.824 ± 0.083
17.1	6.4	20	43.22 ± 4.97	96.4	39.38 ± 3.95	96.7	0.911 ± 0.167
17.0	12.3	5	26.18 ± 1.22	99.5	22.93 ± 0.44	99.9	0.876 ± 0.058
17.0	12.3	20	35.63 ± 3.49	96.5	26.36 ± 3.03	95.0	0.740 ± 0.204
17.1	20.1	5	24.08 ± 1.35	99.0	17.99 ± 0.81	99.3	0.747 ± 0.096
17.1	20.1	20	24.78 ± 3.45	91.4	18.21 ± 2.08	92.8	0.735 ± 0.245
16.9	31.8	5	15.00 ± 0.54	99.7	12.83 ± 0.68	98.4	0.855 ± 0.075
16.9	31.8	20	15.71 ± 0.68	99.0	11.84 ± 0.34	99.5	0.754 ± 0.069

similar to that reported by Silva (1995). It is also of the same order of magnitude as the ratio of CO_2 to O_2 diffusivity in air (0.77–0.83) (Loncin & Merson, 1979; Kays & Crawford, 1980; Perry, Green & Maloney, 1984; Mannapperuma et al., 1989) and thus similar to the ratio between the square root of the molar masses of O_2 and CO_2, according to Graham's law (Atkins, 1990). Therefore, the β ratio in tubes in this range of dimensions cannot be controlled by changing the perforation dimensions.

O_2 and CO_2 mass transfer coefficients were recalculated using the β ratio estimated above. Several equations were tested to relate the mass transfer coefficients to tube dimensions. The best fit was obtained by the following multiplicative non-linear equations (Fig. 2):

$$K_{O_2} = a \times D^b \times L^c, \tag{3}$$

$$K_{CO_2} = \beta \times a \times D^b \times L^c. \tag{4}$$

This model has a functional form similar to the one that would be expected by dimensionless analysis and has the advantage of having less parameters than those suggested by Emond et al. (1991) and Silva (1995).

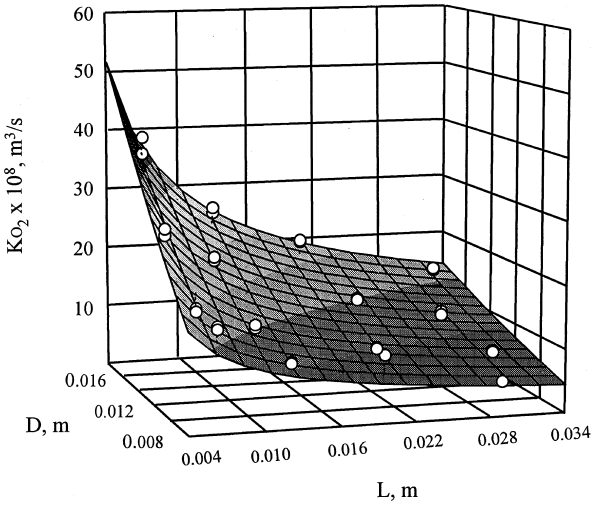

Fig. 2. O_2 mass transfer coefficients determined by application of Eqs. (1) and (3) as a function of tube diameter and length.

In order to improve parameter estimation, Eqs. (3) and (4) were substituted in Eq. (1) for O_2 and for CO_2 respectively, and a single non-linear regression was

Table 3
Parameters of the mathematical model (Eqs. (3) and (4)) and relevant statistical data

$a \pm SD$	$b \pm SD$	$c \pm SD$	R^2
$(6.42 \pm 0.58) \times 10^{-6}$ $m^{2.148}\ s^{-1}$	1.45 ± 0.02 Dimensionless	-0.598 ± 0.008 Dimensionless	97.7%

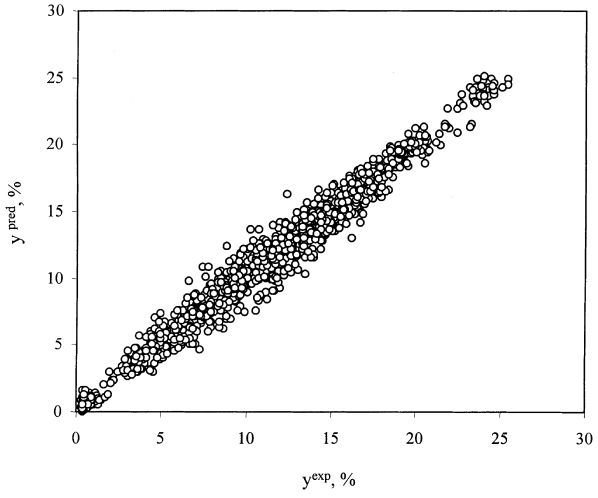

Fig. 3. Relationship between experimental and predicted concentration values for O_2 and CO_2 (data used for model build-up).

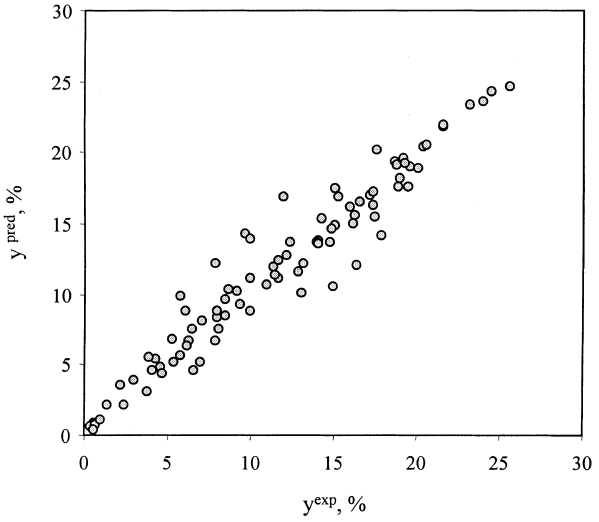

Fig. 4. Relationship between experimental and predicted concentration values for O_2 and CO_2 (data used for model validation).

applied to the whole set of data, estimating the parameters a, b and c as shown in Table 3. The model adequately describes the process, as shown by the high R^2 (97.7%). The values of b and c, the power constants of the tube diameter and length, were, respectively, 1.45 and -0.598. These values are smaller than those expected by Fick's law (2 and –1, respectively), again showing that end effects may play an important role in gas exchange. Fig. 3 presents the fair agreement between predicted and experimental concentration values for O_2 and CO_2, where it is visible that the trend is not biased. The surface on Fig. 2 shows the dependence of the O_2 mass transfer coefficients on tube dimensions, allowing the interactive effects between tube diameter and length to be assessed. The effect of tube diameter is more pronounced for the small length tubes, while the effect of tube length becomes more important as tube diameter increases. Examples of the fit of the overall model to the experimental concentration data are also included in Fig. 1. Results are very close to the fit of the individual model, also indicating the quality of the predictive model.

3.3. Model validation

In order to further test the model, it was applied to predict both O_2 and CO_2 concentration changes in eight additional experimental runs. Fig. 4 shows the fit between predicted and experimental concentration values for O_2 and CO_2, where a fair agreement is clear, thus confirming the predictive ability of the model.

4. Conclusions

The lumped mass capacitance model was confirmed to yield an adequate description of the O_2 and CO_2 exchange through a macro perforation. Temperature had no significant effect on O_2 and CO_2 mass transfer coefficients in the range of temperatures tested. A mathematical model relating O_2 and CO_2 mass transfer coefficients to diameter and length of perforations was developed. This model, coupled with the mass capacitance model was shown to accurately predict the gas exchange, in spite of being much simpler than previously published models. The β ratio was found to be independent of temperature and perforation dimensions.

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