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Determination of O_2 and CO_2 transmission rate of whole packages and single perforations in micro-perforated packages for fruit and vegetables

Q1 Hanne Larsen *, Kristian Hovde Liland

Nofima – Norwegian Institute of Food, Fisheries and Aquaculture Research, PB 210, N-1431 Aas, Norway

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ABSTRACT

Microperforated packages are in widespread use for whole and fresh-cut fruit and vegetables, and there is a need for a simple and cost efficient methodology to accurately determine gas transmission rates for different packages. This work demonstrates a static technique using a low cost gas analyser for determining the O_2 and CO_2-transmission rates and permselectivity for whole perforated and non-perforated packages stored at different temperatures. The work further demonstrated the possibility to calculate the transmission rates for single holes, and results for single perforations agreed well with results in other studies. The O_2- and CO_2-transmission rates in perforated packages were not significantly affected by temperature in the range 5–23 °C, whereas transmission rates increased with increasing temperature for non-perforated packages. Gas transmission measurements can be used within quality control, in the choice of appropriate packaging for different fruit and vegetables and as an important parameter in EMAP modelling.

1. Introduction

Microperforated films are commonly used for modified atmosphere packaging (MAP) of fruits and vegetables with high respiration rates. Different headspace conditions can be achieved in the package depending on the interactions between respiratory activity of the packaged produce and gas transfer through the polymeric matrix and microperforations (Lucera et al., 2011). This technique is often denoted equilibrium modified atmosphere packaging (EMAP). The choice of product optimised film is crucial to obtain optimum modification of the atmosphere and avoid extremely low levels of O_2 and/or high levels of CO_2, which could induce anaerobic metabolism with possible off-flavour generation and risk of anaerobic microorganism proliferation (Beaudry, 2000; Watkins, 2000).

Knowledge of the gas transmission rate of the package is one of the key factors in EMAP modelling, and the permselectivity ratio P_{CO2}/P_{O2}, commonly denoted β, is an important parameter being different for continuous and perforated materials (Beaudry, 2008). The gas exchange in a perforated package occurs almost entirely through the microperforations, and various mathematical models have been proposed in order to describe the exchange of gases through the perforations. The application of Fick's law is widespread, and the modified model of Fishman et al. (1996) is commonly used. Ghosh and Anantheswaran (2001) measured the oxygen transmission rate (OTR) of microperforated films using a static method to compare the experimental data with the results predicted by published models. They found that the modified model based on Fick's law as proposed by Fishman et al. (1996) had very good agreement with the experimental data from the static method used in their study. However, all these mathematical models assume the uniform production of microperforations that are round, within the required size range, and unobstructed (Allan-Wojtas et al., 2008). Hence, if the perforations are irregular in size and thickness, methods for direct measurement of the gas transmission rate in perforated packages can be useful in many situations.

Dynamic or coulometric methods are commonly used for measuring OTR and carbon dioxide transmission rate (CO_2TR) of continuous film and packages. These methods are of doubtful application for perforated materials due to the gas convection taking place when the pressure on each side of the material is slightly unbalanced (Ozdemir et al., 2005). Another disadvantage of the coulometric method commonly used for films and packages, is that this equipment cannot measure gas transmission rate at temperatures lower than 10 °C (Abdellatief and Welt, 2012; Lucera et al., 2011), while the recommended storage temperature for fruit and vegetables is 5 °C or lower. Hence, most experimental systems for measuring the permeability of perforated or microperforated materials are static (Gonzalez et al., 2008). Ghosh and Anantheswaran (2001) used both a static and a flow-through technique to measure the OTR of microperforated films. They stated that the static method better simulates the actual package situation, and found that the repeatability of the static method was better than the flow through method resulting in lower standard deviation

* Corresponding author. Tel.: +47 64970100; fax: +47 64970333.
E-mail address: hanne.larsen@nofima.no (H. Larsen).

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values. However, the disadvantage of the static method is that it takes time to get the results. Generally, few published work are found in literature on direct methods to measure the OTR and CO_2-TR of microperforated films and packages. Ozdemir et al. (2005) measured OTR and CO_2TR in non-perforated and perforated 35 µm polypropylene pouches using a static technique and a gas chromatograph for gas concentration measurements. The measured gas transmission values were calculated for the flat film. Gonzalez et al. (2008) used a static measuring system and a gas chromatograph to measure OTR and CO_2TR of single perforations with different dimensions and thicknesses. Based on the experimental data, they proposed an equation describing the dependence of the transmission rates on the perforation area, where the only input is the perforation size measured by an ocular microscope. Their data predicted by the empirical equation was compared to five other bibliographic models. The O_2 and CO_2 transmission rates predicted by their empirical equation were very close to those obtained with the modified Fick's equation as described by Fishman et al. (1996). However, using the empirical model proposed from Gonzalez et al. (2008) still requires uniform holes in order to calculate the perforation area. Oxygen transmission rate for perforated packages can also be measured using a dynamic accumulation method with inexpensive fluorescence oxygen sensing technology (Abdellatief and Welt, 2012). However, this equipment does not measure CO_2, and hence, the CO_2TR cannot be measured for the packages.

Another factor to be considered when working with perforated packages is the perforation size in relation to the storage conditions (calm or convective). Allan-Wojtas et al. (2008) compared different microscopy techniques to study the microstructure of microperforations in plastic films and relate microperforation microstructure to gas transmission characteristics under calm and convective conditions. They observed a linear increase of both O_2 and CO_2 transmission rates with the area of the holes for microperforations in the range of 30–100 µm, for diffusion under calm conditions. Their study also indicated that microperforations larger than 55 µm can lose their diffusion constant if convection is present, and most consistent OTR results were achieved using numerous small holes rather than fewer large ones.

Although most of the gas exchange in a microperforated package occurs through the perforations, in some packages with a low number of perforations the gas flux will have a combination of transmission through the polymer material and transmission through the perforations (Beaudry, 2008). Measuring on the whole package will take into account and simultaneously measure the transmission rate through the polymer material and the perforations at the conditions of intended storage. Larsen et al. (2000) demonstrated a method for measuring OTR of whole packages denoted the ambient oxygen ingress rate (AOIR) method. This method has many advantages such as: (1) use of low cost equipment compared to other commercial permeation equipment, but still with sufficient accuracy; (2) the method has high capacity and many packages can be measured at the same time at different conditions; (3) the OTR can be measured at most temperatures, including freezing temperatures (Larsen, 2004), whereas many commercial available permeation instruments cannot measure at temperatures lower than 5–10 °C; (4) the method measures on the whole package, including heat seals and other possible defects created under the converting process, and after e.g. thermoforming that stretches the materials giving gas transmission rates different from the flat film; (5) the method can be applied on most kinds of whole packages consisting of different materials, including fibre based materials.

The aim of this work was to further develop the AOIR-method and verify the methodology on perforated packages including the measurement of CO_2TR.

The results with this new alternative and simple low cost method for measuring OTR and CO_2TR for whole packages with and without perforations and the single perforations were compared to other research works. Using the developed method, the influence of storage temperature and the difference in β-values for the continuous and perforated packages was also studied. The O_2 and CO_2 transmission rates were studied using different films with different perforation sizes, perforation methods and amount of perforations.

2. Materials and methods

2.1. Packaging materials, preparation of samples, gas concentration measurements, equipment

2.1.1. Packaging materials and packaging procedure

Three series of high density polyethylene (HDPE) trays (Promens, Kristiansand, Norway) were flushed with the gas mixture 5% O_2, 10% CO_2 and 85% N_2 before sealing with three different top webs using a Polimoon 511VG tray sealing machine (Promens, Kristiansand, Norway).

One series of ten 1500 ml trays were sealed with a 12 µm polyester/40 µm polyethylene (PET/PE) (Amcor Flexibles, Ledbury, England) top web. The top web of all the packages was punctured once with an acupuncture needle before storage at 4 °C (named 'Mech-PET' in the following). The irregular hole made with the needle may simulate the shape of holes made by different mechanically puncturing equipment.

Another series of 1500 ml trays were sealed with an Amcor P-plus 12 µm PET/40 µm PE film (Amcor Flexibles, Ledbury, England) with four microperforations (named 'Micro-PET' in the following). The holes (1, 2, 3 or 4) were accordingly covered with septa just after sealing, creating packages with different transmission rates depending on the amount of perforations. The packages were stored at 5 °C, 10 °C and 23 °C during the sampling period. These series were run with 4 replicates.

A third series of 1100 ml trays were sealed with a 20 µm oriented polypropylene (OPP)/25 µm PE film from Sealed Air (Oslo, Norway) giving a non-perforated package with relatively high gas TR (named 'non-perforated package' in the following). These packages were stored at 5 °C and 23 °C during the sampling period, and were run with 4 replicates.

Pieces (360 × 267 mm) of 25 µm biaxially oriented polypropylene (BOPP) film (ScanStore Packaging AS, Middelfart, Denmark) without perforations and with different number of microperforations (denoted 3000, 4100 and 5000) were sealed on two sides giving pouches (named 'Micro-BOPP' in the following). The pouches were flushed with 5% O_2, 10% CO_2 and 85% N_2 using a tube and the volume of the packages was 1758 ± 99 ml. The pouches (five replicates of each) were stored at 4 °C during the sampling period.

All our samples were stored in Termaks environmental chambers (Termaks, Bergen, Norway) which are developed for accurate temperature control. A low circulation of air inside the chamber is obtained by conduct system which keeps most of the air stream outside the working chamber giving calm storage conditions. The air flow was measured in the range 0.2–0.4 m s^{-1} using a Kimo thermo-anemometer VT100 (Emerainville, France).

2.1.2. Gas sampling and microscopy of perforations

Changes in headspace gas composition during time in the packages were recorded using a CheckMateII O_2/CO_2 -analyser (PBI Dansensor, Ringsted, Denmark). The headspace gas concentration was measured several times during a storage period of 5–7 days.

The perforations on the different materials were cut from the plastic film and mounted on microscope slides using tape. The film was covered with cover glass and examined under a Leica DM6000B Light Microscope (Leica Microsystems GmbH, Wetzlar, Germany) equipped with an Evolution MP Colour digital camera. We used a 10× objective and grey scale images were captured. Images of 6–8 perforations of each type were captured and the vertical and horizontal diameters (μm) were noted.

2.2. Mathematical model, practical considerations and calculations

2.2.1. Mathematical model

OTR and CO_2TR were calculated from changes in volumetric fractions of the gases inside the package over time according to the theoretical framework outlined in several publications (Ghosh and Anantheswaran, 2001; Gonzalez et al., 2008; Larsen et al., 2000).

The O_2 and CO_2 transmission rates can be calculated by the following equation:

$$TR = -\frac{V}{t_f - t_i} \ln\left(\frac{C_{air} - C_f}{C_{air} - C_i}\right) \quad (1)$$

where V is the volume of the package, t_f the time of the final gas concentration measurement, t_i the time of the initial gas concentration measurement, C_{air} the volumetric concentration of gas in the air (0.21 O_2, and 0.03 CO_2), C_f the volumetric concentration of gas in the package at the final measurement, C_i the volumetric concentration of gas in the package at the initial measurement.

Solving Eq. (1) for the final volumetric gas concentration we obtain the equation:

$$C_f = C_{air} + (C_i - C_{air}) e^{-\frac{TR}{V}(t_f - t_i)} \quad (2)$$

enabling prediction of gas concentrations over time based on the initial conditions similar to the prediction curves presented in Larsen et al. (2002).

The equations above can be used for both non-perforated (Larsen et al., 2000) and microperforated packages (Gonzalez et al., 2008) involving no metabolic activities. However, working with non-perforated packages, one should be aware of the "volume increase effect" as described by Moyls (2004). For packages such as polyethylene with high OTR and flushed with N_2, the volume of the package will increase during time due to higher O_2 than N_2 transmission rate. Hence, the total pressure inside the package will be higher than 1 atm, introducing an error into the theoretical framework. This error can be minimised by performing the measurements early in the run, when oxygen pressure varies between 0 and 0.05 atm (Moyls, 2004).

2.2.2. Optimal initial conditions for OTR and CO_2TR estimation

To be able to measure changes in O_2 and CO_2 concentrations due to transmission, one obviously has to have initial conditions in the package differing from the outside air. However, which initial conditions that gives the best OTR estimation needs some consideration. If the initial atmosphere in the package has C_{O2} = 0% and C_{CO2} = 21%, there will be a rapid change in concentrations the first few hours. This means that timing of the measurements to obtain representative results has to be accurate. If, on the other hand, initial concentrations of C_{O2} = 20% and C_{CO2} = 1% are chosen, the change in concentration will be slow. This would require very accurate concentration measurements. A compromise would be much more robust by reducing the need for accurate timing and concentration measurements. Consequently the initial concentrations chosen in the presented work were around C_{O2} = 5% and C_{CO2} = 10%.

The resolution and accuracy of the gas analysing instrument should also be taken into account. The CheckMate-instrument has different resolution and accuracy of the sensors in the various ranges. The zirconia sensor, measuring O_2, the resolution is 0.1% absolute in the range above 10%, 0.01 in the range above 1% and 0.001 in ranges below 1%. The accuracy is ±0.01% absolute in range below 1% and ±1% relative in the range above 1%. The CO_2 infrared sensor resolution is 0.1% absolute with an accuracy of ±0.5% absolute and ±1.5% of reading. Hence, the highest accuracy of the O_2 readings will be in the range below 1% O_2, which is a range being difficult to obtain working with perforated packages where the transmission rate is high and the process is relatively unstable at the start of the measurement period. However, a very high accuracy of the gas transmission rate (gas TR) measurements is, in practical use, not necessary due to the large variation in all the other factors constituting a part of the modelling of optimal packages for fruit and vegetables.

2.2.3. Utilising more than two measurement time points

Calculating transmission rates from two time points alone can be vulnerable to measurement errors and dependent on several parallel tests to ensure stable results. To improve robustness of the calculations, more than two time points can be calculated. However, this does not fit into the existing formulae for transmission rates. A strategy for obtaining transmission rates indirectly in a spread sheet is the following (example using oxygen): (1) Make an initial guess of the OTR in one cell. (2) Make formulae for predicting the oxygen concentration at all included time points having the first time point as initial time and OTR from the given cell. (3) Make formulae for calculating the squared error between measured and predicted concentration and sum these up. (4) Use a solver to find the OTR value minimising the sum squared error.

The strategy described above can be used separately on one and one measurement series or on all series at the same time. The former can be used to find a mean transmission rate and its standard deviation across measurement series, while the latter will be an even more robust estimate of the true transmission rate. As a result, it is possible either to get more reliable results using the same number of parallels as usually done, or it can reduce the number of parallels without loss of accuracy.

Our calculations showed that the most stable and representative values for the gas TR values were obtained when the package was allowed to equilibrate for approximately 1 day before the first gas sampling, and the changes in O_2 or CO_2 concentration should be minimum 2% before the last gas sampling after 2–3 days depending on the type of package. The conditioning time of approximately 1 day before the first gas sampling was especially important for the packages flushed on the tray sealing machine, due to an unstable gas ingress process in the beginning of the test run probably caused by the initial vacuuming of the package in the tray sealer. In packages flushed with the gas mixture using a tube, the gas ingress process was more stable earlier in the test period. A longer storage period for the non-perforated packages was beneficial giving larges differences in CO_2-concentrations at the first and last sampling time reducing the errors in lower accuracy of readings.

3. Results and discussion

3.1. Gas transmission rates for non-perforated and perforated packages stored at different temperatures

Gas transmission for three different types of packages was measured and calculated according to the previous description. The results for both the whole packages and for single perforations are presented in Table 1. The OTR and CO_2TR for the single perforations were calculated by subtracting the transmission rate value for the packaging material (0 perforations) from the transmission rate va-

JFOE 7409
11 June 2013
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Table 1
OTR, CO_2TR values and CO_2TR/OTR ratio for three types of packages with different number of perforations and single perforations measured at different temperatures.

Package	Perforations	Temperature	OTR/pkg (mL d^{-1})	CO_2TR/pkg (mL d^{-1})	Ratio CO_2TR/OTR	OTR/perf. (mL d^{-1})	CO_2TR/perf. (mL d^{-1})	Ratio CO_2TR/OTR/perf.
Mech-PET	1	4	284 ± 20	257 ± 34	0.9	279 ± 19	242 ± 33	0.9
Micro-PET	0	5	5 ± 1	15 ± 3	3.1			
	1	5	103 ± 5	108 ± 5	1.0	98 ± 5	92 ± 5	0.9
	2	5	185 ± 20	172 ± 13	0.9	90 ± 10	78 ± 7	0.9
	3	5	274 ± 17	241 ± 15	0.9	90 ± 6	75 ± 5	0.8
	4	5	366 ± 27	322 ± 26	0.9	90 ± 7	77 ± 7	0.8
Micro-PET	0	10	5 ± 1	19 ± 4	3.7			
	1	10	134 ± 18	124 ± 13	0.9	129 ± 18	105 ± 13	0.8
	2	10	193 ± 7	171 ± 3	0.9	94 ± 4	76 ± 2	0.8
	3	10	279 ± 8	251 ± 7	0.9	91 ± 3	77 ± 2	0.8
	4	10	368 ± 12	329 ± 8	0.9	91 ± 3	77 ± 2	0.9
Micro-PET	0	23	10 ± 1	41 ± 4	4.3			
	1	23	131 ± 22	137 ± 6	1.0	121 ± 22	96 ± 6	0.8
	2	23	224 ± 25	218 ± 28	1.0	107 ± 12	89 ± 14	0.8
	3	23	309 ± 14	295 ± 17	1.0	100 ± 5	85 ± 6	0.8
	4	23	374 ± 21	354 ± 18	0.9	91 ± 5	78 ± 5	0.9
Micro-BOPP	0	4	155 ± 39	267 ± 86	1.7			
	6 or 7	4	745 ± 51	693 ± 84	0.9	88 ±5	63 ± 13	0.7
	11	4	1083 ± 68	1013 ± 22	0.9	84 ± 6	68 ± 2	0.8
	14 or 15	4	1434 ± 137	1229 ± 145	0.9	88 ±6	66 ± 8	0.8

lue of the whole package with perforation. The remaining gas TR value for the perforations was then divided by the number of perforations, giving the gas TR per perforation.

Perforations made by the acupuncture needle had the highest gas TR rate, almost threefold the values for the laser perforations in the PET/PE-film and BOPP-film (Table 1). The perforations in the PET/PE-film had slightly higher gas transmission rates than the perforations in the BOPP-film. Gonzalez et al. (2008) measured OTR and CO_2TR for Amcor P-plus film with different perforation sizes and thicknesses of the films. The calculated area (mean value) for the laser perforations in the Amcor P-plus PET/PE-film in our experiment was approximately 6500 μm^2. The results from Gonzalez et al. (2008) showed that the OTR and CO_2TR for a perforation with an area of 6500 μm^2 were approximately 135 and 115 mL gas d^{-1}, respectively. Our gas TR values for the perforations in the Amcor P-plus film were lower but within the same range (Table 1).

The permselectivity ratio P_{CO2}/P_{O2}, commonly denoted , is different for continuous and perforated materials (Table 1 and Fig. 1). The ratio in our experiment was in the range from 3.1 to 4.3 for Micro-PET, 1.7 for Micro-BOPP without perforations and 3.5 for the non-perforated package with OPP/PE as top web. The permselectivity ratio for different polymeric films can in general vary from 2 to 8 (Beaudry, 2008; Gonzalez et al., 2008; Ozdemir et al., 2005). However, since the packages in our experiment were a combination of HDPE trays with PET/PE and OPP/PE as top webs, and permselectivity values commonly are given for pure materials, it is difficult to find corresponding results to compare with in literature for this package. The ratio of oriented polypropylene is, according to Ozdemir et al. (2005), approximately three depending on its manufacturing conditions. They measured the ratio to 1.94 for 35 µm OPP film in their experiment, which is in the same range as our value of 1.7.

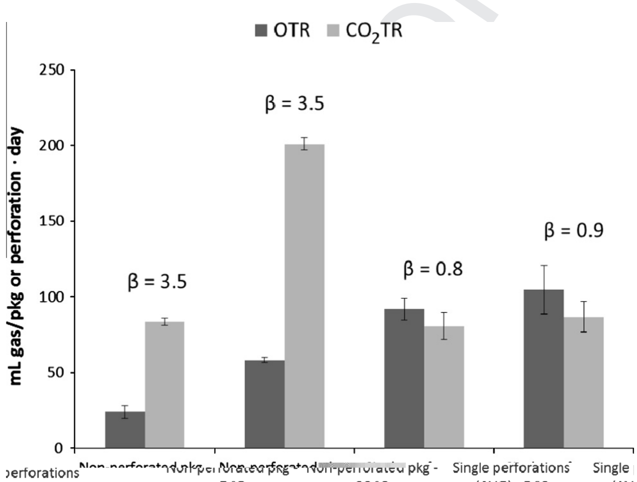

Fig. 1. OTR and CO_2TR in non-perforated packages and single perforations (average values) in Micro-PET packages stored at 5 °C and 23 °C.

The permselectivity for the perforated materials in our study was in the range 0.9–1.0 for whole packages, and 0.8–0.9 for the single perforations (Table 1). Our values are in accordance with the findings of other authors (Fonseca et al., 2000; Gonzalez et al., 2008). Gonzalez et al. (2008) found the quotient CO_2TR/OTR to be 0.89 ± 0.05 for the Amcor P-plus film used in their experiment, and Ozdemir et al. (2005) reported a permselectivity value of 0.87 for one single perforation in the 35 µm OPP film.

The influence of storage temperature on gas transmission rate is also different for continuous and perforated materials (Fig. 1). Storage at 5 °C compared to 23 °C showed no significant differences in gas TR for the average single perforations, whereas OTR and CO_2TR increased by a factor of 2.4 from 5 °C to 23 °C for the non-perforated package (HDPE-tray with OPP/PE top web). The finding for the single perforations is in accordance with the results in the work by Fonseca et al. (2000). They analysed the O_2 and CO_2 exchange rate through a single tube at 5 °C and 20 °C, and found that temperature had no significant effect on O_2 and CO_2 transfer coefficients in this range of temperature. Other experiments on non-perforated packages using the AOIR-method have demonstrated an increase in OTR with higher temperatures (Larsen, 2004). The OTR increases with about 9% per °C for many polymers above the glass temperature (DeLassus, 1997).

3.2. Gas transmission rates for single holes – static, theoretical and exact data

In order to compare our results to other authors' work, the perforations were placed under a light microscope and the mean area for each type of perforation was calculated (using formulae for el-

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lipse). Pictures of the three types of perforations are presented in Fig. 2. Accurate areas for the mechanical perforations were difficult to calculate, due to very irregular shapes of the perforations. Gas transmission rates were accordingly calculated theoretically using the equations described in Fishman et al. (1996) and Gonzalez et al. (2008). Theoretically calculated transmission rates and transmission rates calculated by our static method are presented in Table 2.

Our results using the static method is lower than the theoretically calculated values using equations from Fishman et al. (1996) and Gonzalez et al. (2008) for the micro-perforated materials, whereas the gas TR values for the mechanically perforated film was slightly higher using our static method (Table 2). However, the calculations for the mechanic perforations according to Fishman et al. (1996) and Gonzalez et al. (2008) might be uncertain due to difficulties in accurate calculation of the area for these irregular holes.

In order to study the best fit between the values given in Table 2 to exact O_2 and CO_2 concentrations in whole packages stored over time, prediction curves (selected packages presented in Fig. 3) were made using Eq. (2). The predicted values for the whole package according to the equations by Fishman et al. (1996) and Gonzalez et al. (2008) were calculated by multiplying the OTR and CO_2TR for a single perforation (Table 2) with the number of perforations and adding the permeability value of the whole container without perforations. Measured values (Fig. 3) using the experimental values from the static method gave the best fit for all 4 samples compared to the exact values in the packages. The curves based on the gas TR values measured by the static method were very close to the exact gas concentrations for the two Micro-PET-packages, whereas slightly higher values were predicted using the static method for the Micro-BOPP packages.

The good results obtained using the static method as presented in this work make this method a versatile technique for determining the O_2 and CO_2 transmission rate of whole packages, perforated and non-perforated, and for single perforations for many types of packages stored at realistic storage temperatures. The method uses low cost equipment and is easy to use, and there will be no need for the use of microscopy to study the perforations in order to calculate the perforation areas. A gas analyser is usually available in most packaging facilities, including packaging houses for fruit and vegetables, especially if they perform MAP. The measured gas transmission values can be fit into programs for EMAP modelling for fruit and vegetables, giving accurate values for the gas transmission in the packages. This method can also be useful in the quality control within the packaging facilities, screening the variation in gas transmission in different film production batches.

4. Conclusion

Gas transmission rates were measured for three different types of perforated packages using a static method and a low cost gas analyser. Gas TR in single perforations could also be calculated. Perforations made by an acupuncture needle had the highest gas TR rate, almost threefold the values for the laser perforations in the PET/PE-film and BOPP-film. The permselectivity ratio P_{CO2}/P_{O2} is different for non-perforated and perforated materials. The ratio in our experiment was in the range from 3.1 to 4.3 for Micro-PET, 1.7 for Micro-BOPP without perforations and 3.5 for the non-perforated package with OPP/PE as top web. The permselectivity for the perforated materials in our study was in the range 0.9–1.0 for whole packages, and 0.8–0.9 for the single perforations, which is in accordance with the findings of other authors. No significant difference was found between average values for OTR and CO_2TR for the single perforations in packages stored at 5 °C, 10 °C and 23 °C, whereas gas TR for the package with non-perforated OPP/PE film increased by a factor of 2.4 by storage at 23 °C compared to 5 °C. Comparing our experimental results to theoretical approximations used by other researchers showed that the measured values using the static method gave the best fit with exact values in the packages. The good results obtained using this static method makes it a versatile method for determining the transmission rate of whole packages, perforated and non-perforated, and for single perforations for many types of packages stored at realistic storage temperatures.

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