

RESEARCH ARTICLE

Role of laser micro-perforations on ethylene transmission rate in packaging materials used for fresh produce

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Abstract

Ethylene transmission through the packaging plays a vital role in determining the ethylene concentration inside the fresh produce package. Several investigations describing the oxygen, water vapour and carbon dioxide transmission in modified atmosphere packaging have been studied previously; however, there is a lack of studies on the ethylene transmission rate. Hence, this study aimed to investigate the ethylene transmission through a laser perforated oriented polypropylene packaging film, commonly used in fresh produce packaging. The impact of the perforation diameter (50–150 μm) and film thickness (20, 25 and 30 μm) at a temperature of 25°C on ethylene transmission rate was investigated. The ethylene transmission rate ($k_{\text{C}_2\text{H}_4}$) varied from 6.75 to 10.06 cm^3h^{-1} . It was found to be varied significantly ($p \leq 0.05$), proving its dependency on the selected perforation diameter and film thickness. An increase in the perforation diameter of packaging film increased the $k_{\text{C}_2\text{H}_4}$ exponentially, whereas the film thickness had less influence on the $k_{\text{C}_2\text{H}_4}$. A model based on the perforation diameter and film thickness was employed with an acceptable R^2 of 0.98. The model parameters ($a = 17.44$; $b = 0.35$; $c = 0.14$) determined in this study would be helpful in designing a packaging system for fresh produce that minimise ethylene accumulation.

KEYWORDS

ethylene, fruit and vegetables, MAP, postharvest, storage

1 | INTRODUCTION

An optimum modified atmosphere can be generated inside a package using a packaging film with suitable O_2 , CO_2 and water vapour permeability, considering the produce respiration and transpiration rate. Commonly used films such as oriented polypropylene, polyethylene and polyethylene terephthalate have low permeability to gases and may lead to anoxia in high respiring commodities.^{1,2} Moreover, these also have high water barrier properties, leading to condensation and growth of spoilage-causing microorganisms.

Hence, the generated in-package gaseous concentration in these films may not be ideal.³ In such cases, micro-perforations on the films can provide an excellent solution at a low processing cost.⁴

Micro-perforated films (perforation diameter not more than 200 μm) with adequate dimension and the correct number of perforations can provide excellent gas exchange rates to develop the desired modified atmosphere and prevent water condensation.^{5,6} Micro-perforated films have been reported to increase shelf life and maintain the quality of many fruit and vegetables.^{3,7–10}

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Several factors can impact gas transmission through perforated media, such as perforation diameter and number, film thickness, air flow and temperature.¹¹ Allan-Wojtas et al.¹² reported that the transmission rate for O₂ and CO₂ increased linearly with the individual perforation area in micro-perforated films. In another study, the authors investigated the impact of temperature on gas transmission rates in micro-perforated packages and found it insignificant.¹³ A study on gas transmission rates (O₂ and CO₂) considering different perforation dimensions and film thickness in micro-perforated films was done by González et al.¹⁴ Castellanos et al.¹⁵ studied the water vapour and O₂ and CO₂ transmission through perforated film. Ramos et al.² examined factors affecting mass transfer through perforations (270–750 µm) in oriented polypropylene film and reported that perforation diameter was more significant than total perforation area.

Additionally, researchers reported that the storage temperature and the air velocity around the package were other significant factors. Such studies on gas transmission rate through perforated media allow optimising the number and size of perforations for the desired MAP conditions.¹⁶ Different methods can be employed to perforate the films, such as the electrostatic method hot and cold dies and laser perforations. Among these, laser perforations provide more uniform holes, and smaller holes can be produced than mechanical methods.¹⁷

Among the studies directed at micro-perforated packages, the transmission of gases, O₂ and CO₂, and water vapour through perforations has been widely studied.^{1,5,13,14,18,19} Different models have been used to study the MAP gases transmission through the perforated package,³ but lumped capacity model is the most used and reliable one.¹³ The ethylene permeability for non-perforated films has been determined previously for different types of packaging films^{20–23}; however, the transmission of ethylene through perforated media has not been investigated. Ethylene is a gaseous plant ripening hormone biosynthesised by fruit and vegetables at varying rates. It is one of the crucial components in package headspace that can impact the produce shelf life. Exposure to ethylene can result in accelerated ripening and decay in these commodities. Other adverse effects of ethylene in fruit and vegetables include the development of physiological disorders such as abscission of leaves, degreening, yellowing, increased susceptibility to microbial growth, development of off-flavours, among others.^{24–29} Even in concentrations less than 0.005 ppm, ethylene is highly biologically active.³⁰ Hence, the prevention of ethylene accumulation in the headspace of a package needs attention.

Knowledge of ethylene transmission through micro-perforations can provide essential information on ethylene accumulation inside micro-perforated packages. Because of scarce studies on ethylene transmission through micro-perforated packages, this study was undertaken with the primary aim of studying the ethylene transmission rate through micro-perforations in films. In this study, the influence of the perforation size (50–150 µm) and the thickness of the film (20, 25 and 30 µm) on ethylene transmission rate through laser perforated polypropylene film was investigated.

2 | MATERIALS AND METHODS

2.1 | Experimental setup

The experimental setup comprised a cylinder (2.5 L) and a lidding ring with a hollow area. Packaging film was placed between the lidding ring and the cylinder. An effective area of 453 cm² of the film was available for gas exchange between the inside and outside atmosphere of the cylinder. The lidding ring was connected to the cylinder using nuts and bolts to make the system airtight. A rubber septum was provided at one end of the cylinder to inject/withdraw samples using a syringe. The cylinder was injected with an initial ethylene concentration of 20 ppm. The experiment was carried out at room temperature (25°C). The aluminium weighing boat containing water (10 mL) was kept inside the container to saturate the air up to 100% relative humidity.

The oriented polypropylene (OPP) packaging film with different thicknesses and laser-perforation diameters (micro-perforation) was provided by DanFresh (Lyngbygade 6 g, 8600 Silkeborg, Denmark). Depending upon the preliminary studies, three thicknesses (20, 25 and 30 µm) and three laser perforations of OPP were selected as process parameters for ethylene transmission. The full factorial experimental plan with all possible combinations (9 experimental runs) was performed.

The perforation diameter was measured using the microscope (Zeiss, model: AXIOSTAR plus; Landshut, Germany). As shown in Figure 1, the diameter was measured in two axes, and the average of two axes was represented as the final perforation diameter.³¹

2.2 | Ethylene measurement

The ethylene transmission rate was determined using gas chromatography (GC) (Nexis GC-2030 Shimadzu, Japan) as per the procedure described by Pathak et al.³² with slight modifications. The sample (0.5 mL) was withdrawn through each septum of the cylinders, and ethylene was measured in GC, operating in splitless mode. RT-Q-Bond

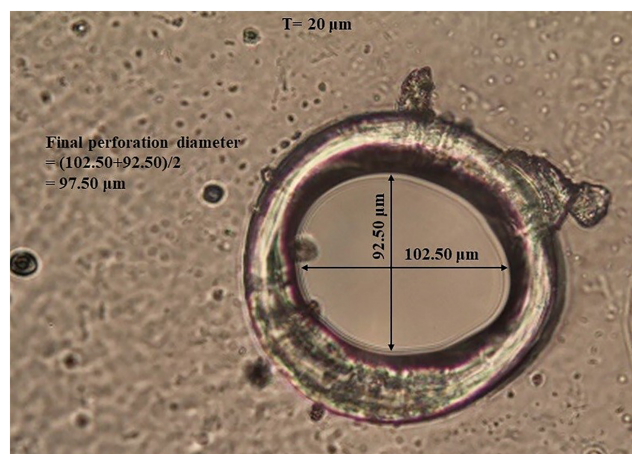


FIGURE 1 Measurement of perforation diameter using a microscope

Alumina column (Shimadzu, Japan) was set at 100°C. During analysis, the flame ionisation detector was operated at 150°C, and the injection temperature was set at 110°C. A carrier gas, helium, was employed at a constant pressure of 120 kPa with a sample flow rate of 8 mL min⁻¹. The detection time of 2.5 min was set for obtaining the chromatograph of ethylene. The software, LabSolutions (Shimadzu, Japan), calculated the area for ethylene gas. It was then compared with the standard area of 10 ppm ethylene (Linde AG, Berlin, Germany) to obtain the actual ethylene concentration in the container. The transmission of ethylene was measured through a complete area of packaging film (considering all micro-perforations formed during manufacturing) and a laser perforation at 25°C with stagnant air over a film (air velocity ~0).

2.3 | Modelling of ethylene transmission

A lumped-capacity model (Equation 1) has been applied to determine the transmission of oxygen and carbon dioxide through perforated packaging materials.^{13,33–36} Therefore, a similar model was applied to estimate the ethylene transmission rate in this study.

$$\ln\left(\frac{y_{ini}-y_{air}}{y_t-y_{air}}\right)=\frac{k_{C_2H_4}}{V_c}t \quad (1)$$

where y_{ini} and y_{air} are the initial (inside the cylinder) and air ethylene concentration, respectively, whereas y_t is the inside cylinder ethylene concentration at time t . A $k_{C_2H_4}$ denotes the ethylene transmission rate, and V_c represents the volume of the cylinder.

The concentration of ethylene in the air was negligible ($y_{air} \sim 0$). So, Equation (1) becomes as follows.

$$\ln\left(\frac{y_{ini}}{y_t}\right)=\frac{k_{C_2H_4}}{V_c}t \quad (2)$$

Further, it can be transformed into an exponential term for modelling and calculation ease.

$$y_t = y_0 e^{-\frac{k_{C_2H_4}}{V_c}t} \quad (3)$$

2.4 | Statistical analysis

Equation (3) was solved in the Statistica (version:10.0) for each experiment to evaluate the ethylene transmission rate ($k_{C_2H_4}$), other model constants and standard error. Duncan test was applied to determine the significance (95% confidence interval) of the ethylene transmission rate at different perforation diameters and film thickness. The effect of film thickness and perforation diameter on ethylene transmission rate was further studied, and a secondary model (Equation 4) was developed.

3 | RESULTS AND DISCUSSION

3.1 | Ethylene transmission rate

As apparent, the ethylene concentration decreased over time for all conditions of OPP film thickness and perforation diameter, as shown in Figure 2. The time required for the equal amount of ethylene transmission through the films was reduced with increased perforation diameter. This trend was observed for each film thickness. The ethylene transmission rate ($k_{C_2H_4}$) varied from 6.75 to 10.06 cm³h⁻¹, and the comparative representation of $k_{C_2H_4}$ with standard errors for all conditions is shown in Figure 3. The highest value of $k_{C_2H_4}$ was obtained at the lowest film thickness and largest perforation diameter and vice versa. The one-way analysis of variance (ANOVA) and Duncan's post hoc test showed that ethylene transmission rates varied significantly ($p \leq 0.05$), as shown in Figure 3.

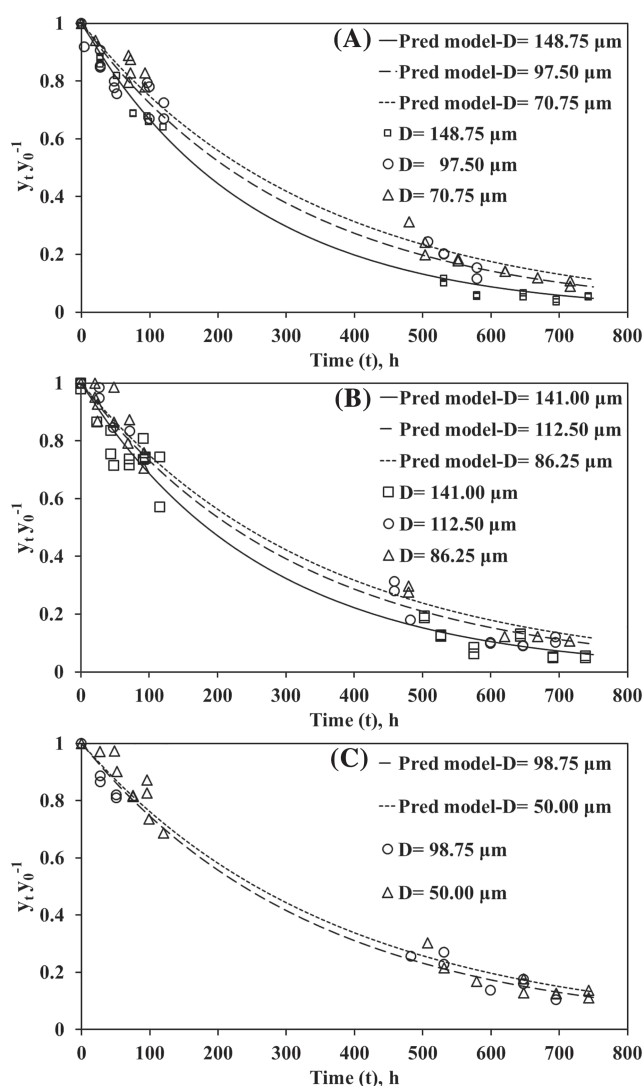


FIGURE 2 Normalised ethylene concentration (y_t/y_0) over time inside the cylinder for OPP film of a thickness (T): (A) 20 µm, (B) 25 µm and (C) 30 µm

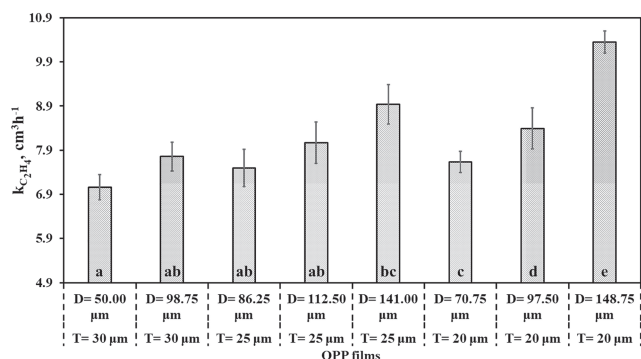


FIGURE 3 Ethylene transmission rate ($k_{C_2H_4}$) for different conditions of perforation diameter (D) and film thickness (T) of OPP films. Bars with different alphabets are significantly different based on Duncans post hoc test at $p \leq 0.05$.

Larsen and Liland¹³ determined the transmission rate of O_2 and CO_2 through micro-perforated ($90.47 \pm 5.29 \mu m$) OPP film (thickness of $20 \mu m$) at $23^\circ C$. The transmission rate of O_2 varied from 3.79 to $5.04 \text{ cm}^3 \text{ h}^{-1}$, whereas for CO_2 , it was from 3.25 to $4.00 \text{ cm}^3 \text{ h}^{-1}$. These O_2 and CO_2 transmission rates are close to the $k_{C_2H_4}$, obtained in this study for similar experimental parameters like static transmission (without any flow of transmission gas), film material, perforation diameter, film thickness and temperature. González et al.¹⁴ also evaluated the transmission of O_2 and CO_2 through micro-perforated (40 – $350 \mu m$) low-density polyethylene (LDPE) with a layer of polyester (thickness, from 29 to $57 \mu m$). The transmission rates of O_2 and CO_2 ranged from 3.13 ± 0.13 ($D = 50 \mu m$; $T = 32 \mu m$) to $13.88 \pm 0.29 \text{ cm}^3 \text{ h}^{-1}$ ($D = 230 \mu m$; $T = 32 \mu m$) and 2.92 ± 0.25 ($D = 50 \mu m$; $T = 32 \mu m$) to $12.08 \pm 0.33 \text{ cm}^3 \text{ h}^{-1}$ ($D = 50 \mu m$; $T = 32 \mu m$), respectively.

The transmission rate for ethylene observed in this study was higher than O_2 and CO_2 for the corresponding perforation diameter and film thickness mentioned in the literature. Thus, the ratios of $k_{C_2H_4}/k_{O_2}$ and $k_{C_2H_4}/k_{CO_2}$ were found to be 2.12 and 2.48 , respectively, for a single perforation (the values of k_{O_2} and k_{CO_2} are taken from Larsen and Liland¹³). The ratio of ethylene permeability to oxygen permeability has been determined by Wang et al.²¹ and was 1.97 for LDPE ($T = 85 \mu m$), whereas 1.52 for HDPE ($T = 40 \mu m$) without any perforation at $19^\circ C$ and $30^\circ C$, respectively. Thus, with a single perforation in OPP film, the ethylene transmission rate was significantly higher than the non-perforated film, as reported in the literature, indicating that the OPP film with a perforation would accumulate less ethylene inside the package and delay the ripening of fruits.

3.2 | Effect of perforation diameter and film thickness on ethylene transmission

The effect of perforation diameter (D) and film thickness (T) on ethylene transmission, that is, numerically ethylene transmission rate

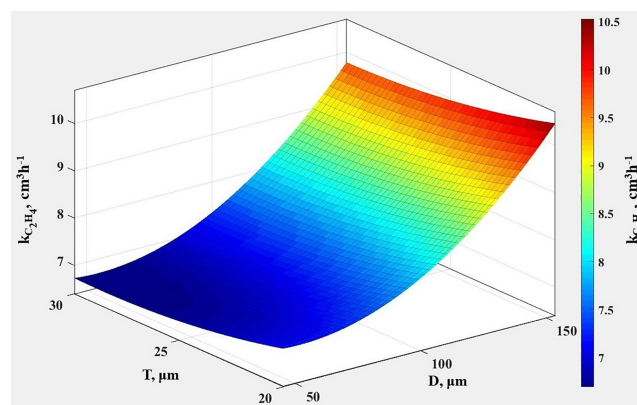


FIGURE 4 Surface plot of the ethylene transmission rate ($k_{C_2H_4}$) for the perforation diameter (D) and film thickness (T)

($k_{C_2H_4}$), is shown using a 3D surface plot (Figure 4). The surface plot with polynomial equation (second degree) and fitting parameters (R^2 , adjusted R^2 , and root mean square error [RMSE]) is shown in Figure 4. The values of R^2 and RMSE were 0.9894 (>0.90) and 0.2245 , respectively, revealing that the model equation of second-degree polynomial was a good fit. Additionally, the adjusted R^2 was considerably close to the R^2 , thus signifying that each term (first and second degree) associated with perforation diameter and film thickness influenced the $k_{C_2H_4}$.

Graphically (Figure 4), it was concluded that perforation diameter increased $k_{C_2H_4}$ exponentially and was a more dominant factor affecting the $k_{C_2H_4}$ than the film thickness. Similar trends were noticed for transmission of O_2 and CO_2 by González et al.¹⁴ when transmission rate was studied as a function of perforation area. As shown in Figure 4, the ethylene transmission rate decreased slightly as the thickness increased at the constant perforation size. This was due to lower convection provided by larger film thickness, consequently, lower mass transfer (diffusion) through the packaging film.³⁷

3.3 | Model development

The transmission rate of a perforated film varied with perforation diameter and film thickness. A decrease in film thickness and an increase in perforation diameter elevated the transmission rate in the current and previous studies.³⁸ Based on this, a model was derived and applied to the ethylene transmission rate of laser perforated OPP films, as shown in the Equation (4).^{39,40}

$$k_{C_2H_4} = a \times D^b \times T^{-c} \quad (4)$$

where a , b and c are the model parameters. The parameter a has a unit of $\text{cm}^3 \text{ h}^{-1}$, whereas b and c are dimensionless.

Substituting Equation (4) in Equation (3) yields Equation (5), which was used to obtain the values of model parameters.

$$y_t = y_0 e^{\frac{a \times D^b \times T - c}{V_c} t} \quad (5)$$

By solving model Equation (5) for comprehensive data, the diagnosis plots were studied to define and validate the developed model. The value of R^2 was 0.9836, showing the best fit. The diagnosis plots (Figure 5) indicated that experimental and model-predicted ethylene concentrations were close to the best fit line ($R^2 = 1$). The maximum number of residues occurred at zero, thus showing that the developed model was well defined and valid for given conditions (perforation diameter and film thickness) of laser perforated OPP films.

The estimated model parameters are shown in Table 1 with the standard errors. The coefficients a and c were insignificant, concluding that they did not affect the $k_{C_2H_4}$, whereas coefficient b was significant, directly affecting the $k_{C_2H_4}$. As concluded earlier from the surface plot, it was confirmed numerically that perforation diameter played a vital role. Therefore, it is considered the most crucial process parameter in designing the MAP for storing fruit and vegetables. Perforation diameter was also reported to be the dominant factor in O_2 transmission through perforated films.²

The developed model would be helpful in designing the MAP for high ethylene producing and ethylene sensitive fruit and vegetables. The perforation size (number of perforations and perforation diameter) could be optimised for specific fruit depending upon its ethylene production rate using this model in such a way that the ethylene accumulation inside MAP would be zero, and the ethylene production rate by fruit and vegetables would be equal to the ethylene transmission

rate through perforations. Ethylene production by fruit and accumulation inside the package and transmission through perforation would be modelled into differential equations and solved to simulate ethylene evolution inside the package. The ethylene transmission rate through perforations may also be affected by temperature, airflow around the package, relative humidity inside and outside the package and molecule interaction of ethylene with O_2 and CO_2 . Thus, it becomes necessary for the future perspective to study the effect of all these conditions on the ethylene transmission rate.

4 | CONCLUSIONS

The dependency of ethylene transmission through laser perforated OPP films on perforation diameter and film thickness has been studied. It was concluded that perforation diameter showed a significant effect on ethylene transmission, measured as an ethylene transmission rate ($k_{C_2H_4}$), ranging from 6.75 to 10.06 $cm^3 h^{-1}$ for perforation diameter (50.00–148.75 μm) and OPP film thickness (20–30 μm). The ethylene transmission rate for all conditions of OPP films varied significantly ($p \leq 0.05$), hence proving its dominion on perforation diameter and film thickness. The polynomial equation (second degree) of the surface plot with R^2 of 0.99 showed an exponential increase in the $k_{C_2H_4}$ when perforation diameter increased, and film thickness slightly affected the $k_{C_2H_4}$ value but had a considerable effect. The model developed for the ethylene transmission rate was acceptable with $R^2 = 0.98$. The model parameters ($a = 17.44$; $b = 0.35$; $c = 0.14$) would be helpful in optimising the perforated films (number of perforations, perforation diameter and film thickness) in modified atmospheric packaging of fresh produce.

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CONFLICT OF INTEREST

The authors declare that they do not have any conflict of interest.

DATA AVAILABILITY STATEMENT

Research data are not shared.

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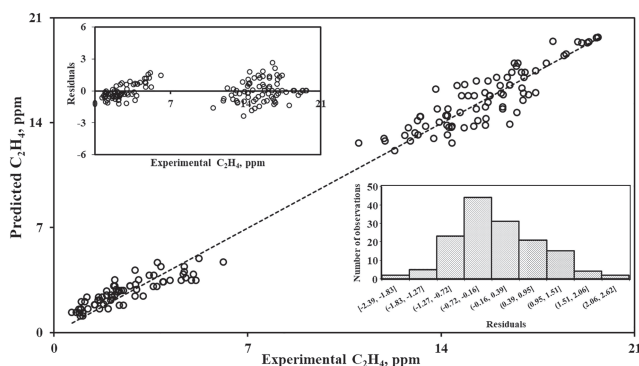


FIGURE 5 Diagnosis plots—predicted C_2H_4 against experimental C_2H_4 , residuals versus experimental C_2H_4 and the number of observations versus residuals for the developed model

TABLE 1 Estimation of model parameters

Model parameter	Estimated values \pm standard error	R^2
a ($cm h^{-1}$)	$17.44 \pm 13.69^*$	0.98
b (dimensionless)	$0.35 \pm 0.05^{**}$	
c (dimensionless)	$0.14 \pm 0.11^*$	

* $p > 0.05$, insignificant.

** $p \leq 0.05$, significant.

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