Aroma Compound and Water Vapor Permeability of Edible Films and Polymeric Packagings

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The permeability of water vapor and aroma compounds through low-density polyethylene packagings and edible films was measured with a dynamic method coupled with a gas chromatograph. Volatile fluxes were analyzed and quantified with two detections: thermal conductivity and flame ionizing detectors. The effects of overpressure and aerodynamic conditions inside the permeation cell were determined, and vapor concentration differentials were only used to measure permeability. Water vapor and aroma permeabilities increase with the penetrant sorption within the film, particularly for hydrophilic films such as cellophane or edible films. Whatever the nature of the penetrant, polar (water vapor) or apolar (1-octen-3-ol), the behavior of edible films and the activation energy of permeation are identical. However, aroma transfer through methylcellulose-based films strongly increases with the water vapor transfer rate (WVTR) due to the plasticization of the polymer network by water. WVTR directly depends on the hydrophobicity of the polymer, but not for 1-octen-3-ol flux.

Keywords: Water vapor; 1-octen-3-ol; permeability; polyethylene and edible films; methylcellulose; gluten

INTRODUCTION

One quality criterion of a foodstuff is aroma, the mixture of volatile organic compounds with low molecular weight. To prevent flavor modification of food products, the transfer of aroma compounds can be limited by the use of appropriate packaging (Mohney *et al.*, 1988).

Several phenomena can happen during the storage of food products such as the "scalping", *i.e.*, food flavor sorption at the inner surface of polymer packaging, or migration of components through the film caused by sorption at the upper side, diffusion within the polymer, and desorption at the down side. This transfer can be oriented from the food product to the surrounding medium ("out migration"), corresponding to a loss of aroma quality, or in the opposite direction ("in migration"), causing a flavor adulteration of foods.

With the development of apolar plastic films used in food packaging such as polyolefins, many studies have dealt with migration of small molecules between foodstuff and wrapping. If the solubility coefficient and diffusivity of volatile compounds in common polymeric packagings are known, the loss of aroma intensity in foodstuffs can be predicted and reduced during storage.

One case often studied is the interaction between aroma compounds from fruit juice such as orange, lemon, or apple and aseptic or film packaging such as low density polyethylene (Sadler and Braddock, 1990, 1991), copolyester and ethylene vinyl alcohol (Baner *et al.*, 1991), or high-density polyethylene and polypropylene (Charara *et al.*, 1992). Most of these works have studied the effects of aroma concentration of solution, medium nature, temperature, and other operating conditions on their sorption within polymer packagings (Nielsen *et al.*, 1992; Sadler and Braddock, 1991). Charara *et al.* (1992), Konczal *et al.* (1992), and Letinski and Halek (1992) studied the influence of film characteristics as structure or crystallinity on aroma sorption.

To improve the efficiency of foodstuff wrapping, the use of edible films and coatings has potential. These offer some advantages over traditional polymeric packagings such as the use of nonpolluting and recyclable raw materials. Coatings have been employed as a preservation method for a long time, such as the coating of fruits with waxes, meat with fats, and candies with sugar or chocolate. The advantages of these are their limitation of solute transfer, not only between food and surrounding media but also between two parts of a composite food (Guilbert, 1986).

Functional properties of edible barriers depend on the film-forming technique used and on the main mechanisms responsible for food deterioration (Kester and Fennema, 1986). Many food macromolecules such as proteins and carbohydrates could be used as edible film forming bases. Until now, most work on small molecules transferred through edible films concerned water vapor transport (Gennadios and Weller, 1991; Martin-Polo *et al.*, 1992a,b; Debeaufort *et al.*, 1993). Edible polymers are mainly hydrophilic, and sorption of aroma compounds is reduced compared to that of apolar plastic packagings. Thus, aroma permeability of edible films should be smaller than that of polymeric films. The aroma barrier of traditional packaging could be improved with edible coatings.

The objective of this research was to better understand water and aroma vapor transfers through edible films. To target this, a dynamic method was developed to measure, simultaneously, water and flavor vapor fluxes.

MATERIALS AND METHODS

Aroma Compound. The volatile selected was 1-octen-3ol (Fluka, Chemika-Biochemika) with the following physicochemical characteristics: mushroom smell; formula $CH_3(CH_2)_4$ -

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Figure 1. Dynamic system to measure vapor and gas permeability through films.

CHOHCH=CH₂; purity, 98%; molecular mass, 128 g mol⁻¹; density at 20 °C, 0.837 kg L⁻¹; solubility in water at 25 °C, 2400 ppm (v/v); saturated vapor pressure at 30 °C, 0.46 mmHg.

The hydrophobicity constant, log P, is 2.6 and was calculated according to the Rekker method (Rekker, 1977). It is typical of a hydrophobic compound (log P > 2).

Packaging Films. A 20 μ m thick cellophane 300P was purchased from Courtauld's Films (U.K.), and a 25 μ m thick low-density polyethylene Riblene FF30 (LDPE) was provided by EniChem Polymères France S.A. (France). Densities are, respectively, 1200 and 846 g L⁻¹ at 25 °C.

Ēdible Barriers. Two kinds of film were prepared from different film-forming substances: methylcellulose, a hydrosoluble cellulose derivative with average molecular mass of 20 000 Da corresponding to 110 glucose units, 40 mPas viscosity of a 2% solution at 25 °C, and 27.5-31.5% degree of methyl substitution (Benecel M021, Aqualon, France); and gluten, a wheat fraction containing at least 75% protein (Manito, Eurogerm, France) with molecular mass between 30 000 and 120 000 Da. All edible films contained 30% with respect to dry basis of polyethylene glycol 400 (PEG400, Merck, Germany) used as plasticizer.

Preparation and Casting of Edible Films. Methylcellulose films (MC) were obtained by solubilization of 4.25 g of methylcellulose in 75 mL of a water-ethanol solution (3:1 v/v)at 75 °C under magnetic stirring for 10 min. Afterward, 1.4 mL of PEG400 was added and the viscous solution was kept for 5 min under the same conditions. The solution was spread out using a thin layer chromatography spreader onto glass plates covered by poly(vinyl chloride) to facilitate the unsticking of films. Films were dried at 25 °C and 40% relative humidity for 12 h.

Gluten films were obtained by dissolving 3.75 g of gluten in 50 mL of a water-ethanol mixture (2:3 v/v) at 60 °C under magnetic stirring. After 5 min, solubilization was completed by homogenizing the suspension at 20 000 rpm for 1 min. pH was adjusted to 4 with 2 N acetic acid and kept constant; 1 mL of PEG400 was added to the suspension, and the mixture was homogenized again. Gluten films were spread out and dried using the same techniques and conditions as for methylcellulose films. Thicknessess of edible barriers were between 20 and 40 μ m after drving.

Vapor Permeability Measurements. A dynamic measurement method of water and aroma vapor fluxes through films or membranes was perfected. The permeation cell was composed of two chambers divided by the film to be studied (Figure 1). The film area exposed to transfer was 15.9 cm². The two chambers were continuously swept by a 30 mL/min helium flow. The aroma and water concentrations in the vapor phase on the upper side of the cell were obtained by mixing three flows: one containing the volatile compound, the second water vapor, and the latter dry helium. Flows containing vapors were obtained from bubbling dry helium through pure compounds (Figure 2). One milliliter of the outgoing flow from the down-side chamber was injected at specified time intervals with an automatic valve in a gas chromatograph. The automatic valve involves an overpressure in the down-side chamber of the cell, and then to equilibrate total pressure on the two faces of the film, a water column was placed at the



Figure 2. Permeation cell.

exit of the upper chamber. In this case, transfers are only due to concentration differential forces.

Water vapor flux was analyzed with a thermal conductivity detector (TCD), and volatile organics were analyzed with a flame ionizing detector (FID). These two detectors were connected in series. Samples were weighed before and after permeation measurements to determine the amount, Q, of volatile sorbed within the films. Q_w and Q_{oct} , are respectively, the amounts of water and 1-octen-3-ol sorbed and are expressed as milligrams of volatile per gram of dry film (mg/g df).

Films were equilibrated at 0 or 53% relative humidity at 25 °C before permeability determinations. Permeation measurements were carried out at 35 or 25 °C, and vapor concentration differentials were between 0 and 21 μ g/mL He for water and between 0 and 5.7 μ g/mL He for 1-octen-3-ol.

Gravimetrical Method. For comparison and validation of the previously described method, transfer rates were also determined with the more traditional gravimetric method AFNOR NF H00-030 (1974) modified, which is a standardized technique for the determination of water vapor transfer rate (WVTR) through sheet packaging materials [homologous to ASTM E96-80 (1990)]. Films were fixed between two Teflon rings on the top of a glass cell as previously described by Debeaufort et al. (1993). The bottom of the cell contained either pure water or a saturated salt solution of known water activity (KCl, $a_w = 0.84$) or pure aroma compound. Test cells were stored in a desiccator over P2O5 with relative humidity close to zero. These test cells were periodically weighed. When the relationship between weight loss and time was linear, the slope of the plot was used to calculate the vapor transfer rate.

Measurements were carried out under the same conditions as the dynamic flow method.

Apparatus. Analyses were carried out on a Chrompack CP 9000 gas chromatograph with thermal conductivity and flame ionizing detectors connected in series and a stainless steel Carbowax 20M column packed with chromosorb W-AW (100-120 mesh). Operating conditions were as follows: helium carrier gas at 30 mL/min; temperature at injection port (190 °C), column (130 °C), and detectors (200 °C). A Shimadzu CR3A recorder-integrator was used.

Calculations. The water vapor transfer rate (WVTR) and aroma flux (F_{oct}) were defined as the ratio of the weight of permeated vapors (g) to the product of area exposed (m^2) and time (s). WVTR and F_{oct} were expressed as g m⁻² s⁻¹.

For each permeability measurement, at least three replicates were made. Differences among data means were tested for statistical significance at the p < 0.05 level using the



Figure 3. Influence of the carrier gas flow on the pressure inside the permeation cell.

Student-Newman-Keuls *t*-test of SAS (Statistical Analysis System, 1991).

RESULTS AND DISCUSSION

Effect of Operating Conditions on Water and Aroma Vapor Transfers. It is well-known that the concentration boundary layer exists on a membrane surface and decreases the efficiency in separation processes, particularly in liquid media. Although a large number of studies on gas permeation or separation with membranes have been reported, there have been only a few papers that examine the boundary layers phenomenon (Haraya *et al.*, 1987). Gennadios *et al.* (1994) have shown that the existence of a stagnant air layer on the surfaces of hydrophilic edible films could result in significant resistance to water vapor transfers. Most of the work on the water vapor permeability of edible films or on the aroma permeability of plastic packagings does not take into account limit layers.

Aerodynamic conditions used to recover permeated volatiles have a strong effect on film permeabilities (Figure 3). The overpressure in the down-side chamber of the permeation cell increases with the downstream flow. A linear relationship is observed between overpressure and flow in the down-side chamber of the cell.

The total pressure differential is in the direction opposite to the concentration gradient. For a constant concentration gradient, vapor transfer decreases with increasing flow in the down-side chamber if any water column is used to balance the pressure in the two chambers of the permeation cell. Pressure differential involves a resistance to aroma and water vapor transfers.

Moreover, even if the total pressure is balanced, the downstream flow strongly affects aroma and water vapor transfer rates (Figure 4). The same behavior was observed for water vapor or aroma compound on methylcellulose, low-density polyethylene, or cellophane films. It seems that the resistance of limit layers, displayed in Figure 4, became negligible for downstream carrier gas up to 30 mL/min. Lamer (1993) showed the same evolution of 1-octen-3-ol fluxes through silicone membranes used in a pervaporation technique. Limit layers were then displayed on the down-side surface of the membrane in the pervaporation modulus.

In the following studies, the helium upstream and downstream in the test cell was always fixed at 30 mL/min.



Figure 4. 1-Octen-3-ol permeability of methylcellulose films vs carrier gas flow ($\Delta C_{\text{oct}} = 5.68 \ \mu \text{g/mL}$ He at 35 °C).

Table 1. Water Vapor Transfer Rate of 20 μm Thick Cellophane Films at 25 $^\circ C$

| method | rel humidity differential (%) | $\frac{WVTR}{(10^{-4}~g~m^{-2}~s^{-1})}$ |
|---------------|----------------------------------|--|
| gravimetrical | 0-85 | >59.7 ^a |
| - | | 61.6 ± 5.0^{b} |
| dynamic | 0 - 85 | 62.2 ± 2.6 |
| | 0-70 | 50.0 ± 2.5 |

^a Chomon (1992). ^b Mean \pm standard deviation.

Table 2. 1-Octen-3-ol Fluxes Measured with the Gravimetrical Method and the Dynamic Method (35 °C, $\Delta C_{oct} = 0-5.68 \ \mu g/mL$ He)

| film | thickness | gravimetric | dynamic |
|---|----------------------|--|--|
| | (µm) | method | method |
| polyethylene cellophane methylcellulose gluten | 25 20 20 35 | $419.4 \pm 21.7^{a} \ { m nd}^{b} \ 713.9 \pm 38.4 \ 158.7 \pm 52.3$ | $\begin{array}{c} 406.1\pm5.2^a\\ <0.06\\ 633.7\pm6.4\\ 190.3\pm22.8\end{array}$ |

^a Mean \pm standard deviation. ^b nd, not determined.

Methodology Validation and Technical Performances. To verify the validity of the dynamic measurement method, water and 1-octen-3-ol transfer rates were determined for each film and compared to results obtained from standardized weight methods.

WVTR through cellophane films was measured at 25 °C to be compared to previous works (Table 1). 1-Octen-3-ol fluxes were determined with the two methods at 35 °C with a saturated atmosphere of pure aroma compound corresponding to a 0-5.68 μ g/mL helium concentration differential (Table 2).

Results obtained by the dynamic method are not significantly different from those determined with gravimetrical methods and in the literature. Ambient temperature being lower than the desiccator's, 1-octen-3-ol condenses on the in-going surface of film when cells are weighed, and this involves an increase of aroma transfer rate. These could explain the lower accuracy of the gravimetrical method for the measurement of aroma fluxes. Moreover, the weight method does not allow one to detect a 1-octen-3-ol flux lower than 36×10^{-6} g m⁻² s⁻¹.

For the simultaneous determination of water vapor and aroma transfers, dilute solutions of aroma compound cannot be used to fix the volatile concentration in the upstream flow. Indeed, with very low permeability films or small concentration differentials, a stationary state of transfer is reached after more than

Table 3. Volatile Fluxes (WVTR and F_{oct}) and Amount Sorbed (Q_w and Q_{oct}) within Tested Films (25 °C, $\Delta C_w =$ 24.7 μ g/mL He, $\Delta C_{oct} = 1.5 \mu$ g/mL He)

| film | $\frac{\text{WVTR}}{(10^{-4} \text{ g})}$ $m^{-2} \text{ s}^{-1}$ | Q _w (mg/g dry film) | F_{oct} (10 ⁻⁶ g m ⁻² s ⁻¹) | Q _{oct} (mg/g dry film) |
|-----------------|---|--------------------------------------|--|--|
| cellophane | 72.78 | 141 | <0.06 | 1.2 |
| methylcellulose | 53.61 | 60 | 219.0 | 100 |
| gluten | 30.27 | 60 | 8.3 | 58 |
| low-density | <0.3 | <0.1 | 91.4 | 4.6 |

8 h which involves an exhaustion of solution. Aroma and water vapor concentrations in the upper-side chamber of the permeation cell cannot be independently changed. These are reasons the use of several dry carrier gas flows bubbling through pure aroma compound or water was chosen. It permits a very large possibility of volatile and water concentrations in the vapor phase and kept them constant in time.

The dynamic method is validated compared to the usual gravimetrical method with a better accuracy and more rapid stationary flow determination. However, it is necessary to sweep the surface of test films with sufficient flow of carrier gas (30 mL/min) to suppress the limit layers resistances.

Effect of Concentrations on Volatiles Transfers. Amounts of penetrant sorbed at the steady state of transfer and water and 1-octen-3-ol fluxes are shown in Table 3. These were determined independently. The behavior of films depends on the nature of volatile, polar or apolar, such as 1-octen-3-ol. Water or hexane vapor sorptions and contact angle measurements between the surface of films and water droplets allow classification of films according to their hydrophobicity: cellophane < methylcellulose and gluten edible films < low-density polyethylene.

Water vapor transfer rate follows the hydrophilicity of films and amount of water sorbed. Although methylcellulose and gluten films sorbed the same amount of water and hexane, *i.e.*, their hydrophobicities are not significantly different, water vapor transport through gluten films is 25% lower than through methylcellulosebased films. Differences among these edible films could be explained by the structures: a rubbery state for methylcellulose films and glassy for gluten at 6% water content and 25 °C (Gontard et al., 1992; Koelsch and Labuza, 1992). Above the glass transition, diffusivity and solubility strongly increase, and thus the permeability increases. Moreover, the greater is the initial water content and at steady state of transfer, the more the water vapor permeability increases (Debeaufort et al., 1994).

No direct relationship is observed between aroma compound fluxes and hydrophobicity of films, but as shown for water vapor, 1-octen-3-ol transfer strongly depends on the amount of volatile sorbed within the film at the steady state of transfer. For hydrophilic polymers (cellophane and edible films), 1-octen-3-ol transfer increases exponentially with the amount sorbed as following (obtained from four edibles films and cellophane): $\ln F_{\rm oct} = 0.085Q_{\rm oct} - 16.66 (r^2 = 0.994 at p < 0.05)$, where $F_{\rm oct}$ and $Q_{\rm oct}$ are expressed as g m⁻² s⁻¹ and mg/g dry film, respectively. It seems that in hydrophobic films such as low-density polyethylene, 1-octen-3-ol fluxes are more influenced by the amount sorbed than in hydrophilic films, because for the same aroma concentration in the film at steady state, transfer rates are 20 times greater.

Interaction between Aroma and Water Vapor Transfers through Methylcellulose Edible Films.



Figure 5. WVTR through methylcellulose films vs water vapor concentration differentiaal at 25 °C (thickness = $30 \,\mu$ m).



Figure 6. 1-Octen-3-ol flux through methylcellulose films vs aroma vapor concentration differential at 35 °C (thickness = $30 \ \mu m$).

The 1-octen-3-ol or water vapor transports through methylcellulose-based, edible films versus volatile concentration differential in vapor phase are similar. These measurements were dealt with independently (Figures 5 and 6). WVTR and F_{oct} increase linearly with the concentration differential above a value between 1.5 and $2 \,\mu$ g/mL He. The nonlinear portion of the plot could be explained by a plasticization effect of penetrant concentration within the film. Transfer rate, determined at several temperatures and constant concentration differential, allows one to calculate the activation energy of tranfer from the Arrhenius equation. These are, respectively, 46 and 60 kJ/mol for water and 1-octen-3-ol fluxes. These positive values for aroma compounds confirm that methylcellulose films are dense (Debeaufort et al., 1994).

Permeability measurements made independently exhibit the same order of magnitude for activation energy and similar behavior of water vapor and 1-octen-3-ol permeation process. However, WVTR strongly affect the aroma compound flux through methylcellulose-based films as displayed in Figure 7. Indeed, the 1-octen-3-ol transfer rate increases exponentially with increasing WVTR. This phenomenon is mainly due to a great plasticization of the methylcellulose matrix by water content which increases with WVTR and also to a sweeping of the aroma compound with water. Aroma and water vapor fluxes are synergistic. The increase of volatile flux with the flux or the sorption of another vapor was observed by several authors. Indeed, the exponential increase of vapor or gas (O_2, CO_2) transfer



Figure 7. 1-Octen-3-ol transfer rate profile curves through methylcellulose films as a function of the water vapor transfer rate at 35 °C (constant aroma concentration differential = $0.82 \mu \text{g/mL He}$).

with water content or relative humidity (Letinski and Halek, 1992; Chomon, 1992), or of oxygen transfers with limonene concentration (Sadler and Braddock, 1990, 1991) within several polymeric packagings such as nylon 6 or polyethylene, was observed. All of them explained these phenomena by a plasticization of the polymer matrix by the penetrant.

In summary, the methodology developed in this work allows measurement of the fluxes of organic volatiles, water vapor, and noncondensable gases through plastic films as well as through edible films.

Aroma fluxes through edible films are water vapor sensitive, but their behaviors are similar for water vapor and 1-octen-3-ol. Gluten films exhibit aroma permeability 10 times lower than those of polyethylene packagings. Thus, their use for cheese wrapping, such as for camembert cheese, seems very interesting to reduce mushroom off-flavors in refrigerated boxes during storage. Moreover, the very high water vapor transfer allows the respiration of fungi. These films could be applied on the surface of the traditional cheese packaging or used alone as a biodegradable film.

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