

Gas Permeability of Fruit Coating Waxes

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Abstract. The permeability to O₂, CO₂, C₂H₄, and water vapor was determined for 19 commercial fruit wax coatings, four ingredients thereof, and one shrink-wrap film. For the commercial coatings, the O₂ permeability at 50% relative humidity and 30C ranged from 470 to 22,000 ml (STP) × mil/(m² × day × atm) (1 mil = 0.0254 mm) with CO₂ permeability two to eight times as high. Permeability to noncondensable gases tended to be higher for coatings made from carnauba wax than for those made from shellac and rosin. Commercial fruit wax had sufficiently low noncondensable gas permeability to account for large reductions in the respiration rate of coated fruit. Wax coatings could be improved if permeability were controlled:

Coatings applied to the surfaces of fruits and vegetables are commonly called 'waxes, whether or not any component thereof is actually a wax. Commodities that are waxed include apples, avocados, citrus, cucumbers, eggplant, peaches, sweet peppers, and tomatoes. Waxing improves appearance, but waxes are often selected with little consideration for other properties.

The extensive literature documents various properties that are altered by the waxing of fruits and vegetables. Hardenburg's bibliography (1967) covers 292 papers on waxing. Since then, further studies have described the effects of waxing on diverse crops.

Perhaps the most-studied property of waxed fruit is its weight loss during storage (Bramlage, 1986; Cohen et al., 1990; Cuquerella et al., 1981; Erbil and Muftugil, 1986; Farooqi et al., 1988; Hasegawa et al., 1981; Krishnamurthy and Kusalappa, 1985; Lidster, 1981; Paull and Chen, 1989; Purvis, 1983; Tewari et al., 1980; Wells, 1973). In almost all cases, waxed commodities lost weight more slowly than unwaxed controls. In fact, weight reduction has been recommended as a criterion of good waxing (Hall, 1981; Tugwell, 1980).

Waxing also has been studied in relation to spoilage, especially chilling injury and browning. In many of these cases, waxed fruit had less spoilage and a lower respiration rate than uncoated samples. Prevention of spoilage was sometimes attributed to adjuncts, such as fungicides or bioregulators, but more often to the diffusion barrier formed by the coating. The barrier hindered O₂ and CO₂ diffusion, thus reducing the respiration rate (Banks, 1984a, 1985; Erbil and Muftugil, 1986; Farooqi et al., 1988; Meheriuk and Porritt, 1972; Smith and Stow, 1984; Waks et al., 1985). Another benefit of waxing is retention of firmness. Waxing was usually firmer than the controls. Coatings also prevent spoilage by serving as a barrier to water vapor (Grierson and Wardowski, 1978; Morris, 1982). The reduction in spoilage is of such significance that waxing is considered a cost-effective substitute where refrigerated storage is unaffordable (Dalal et al., 1971).

A principal disadvantage of wax coatings is the development of off-flavors from their use (Ben-Yehoshua, 1967; Chen and Paull, 1986; Cohen et al., 1990; Cuquerella et al., 1981; Dhalla

and Hanson, 1988; Erbil and Muftugil, 1986; Farooqi et al., 1988; Krishnamurthy and Kusalappa, 1985; Paull and Chen, 1989; Tewari et al., 1980). Adverse flavor changes have been attributed to the inhibition of O₂ and CO₂ exchange, thus resulting in anaerobic respiration and elevated ethanol and acetaldehyde contents (Ahmad and Khan, 1987; Banks, 1984a; Cohen et al., 1990; Cuquerella et al., 1981; Drake et al., 1987; Nisperos-Carriedo et al., 1990; Risse et al., 1987).

In general, with the exceptions of appearance and lubrication (Lidster, 1981; Mellenthin et al., 1982), the literature shows that the effects of waxing are directly related to gas exchange between the fruit and its environment. However, the same literature provides no values for the permeability properties of fruit coatings except for a few estimates made from permeance of waxed vs. unwaxed fruits (Banks, 1984a; Ben-Yehosua et al., 1985; Paull and Chen, 1989) or based on relative values from storage of the same commodity with different coatings (Ben-Yehosua, 1967; Cuquerella et al., 1981; Hasegawa et al., 1981; Namesny and Decoud, 1988; Rohrbach and Paull, 1982; Siade et al., 1977; Trout et al., 1953). Because of general lack of data on permeability, the considerable literature on coatings is of little value in predicting whether a coating used for one purpose is suitable for another.

We therefore determined values for permeability for some typical fruit wax coatings and components and attempted to show how a coating's permeability may be used to predict its performance.

Materials and Methods

Fick's law. Gas exchange between a plant and its environment is generally governed by Fick's law (Nobel, 1974), which states that flux of a gas or water vapor through a barrier varies with permeance (P) and pressure gradient.

$$\text{Flux} = P \times [\text{pressure difference}] \quad [1]$$

Permeability is defined as

$$\text{Permeability} = P \times \text{thickness.} \quad [2]$$

For barriers in series, such as a laminated film, the permeance through the series (P_s) is related to the permeance of the peel (P_p) and coating (P_c) as follows (Crank, 1956):

$$1/P_s = 1/P_p + 1/P_c. \quad [3]$$

American Society for Testing Materials-recommended metric

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Abbreviations: RH, relative humidity; STP, standard temperature (0C) and pressure (1 atm).

units for permeability are ml (STP) \times mil/(m² \times day \times atm) where 1 mil is 0.001 inch (0.0254 mm). Corresponding units for permeance are ml (STP)/(m² \times day \times atm), which are the units used for permeability of O₂, CO₂, and C₂H₄. The units for water vapor are g mil/m²·day⁻¹·mm⁻¹ Hg for permeability and g·m⁻²·day⁻¹·mm⁻¹ Hg for permeance. Nevertheless, for the calculation of the ratio of water vapor to O₂ permeability (see Table 4), both first had to be converted to the same units. For that conversion, 1 g mil/m²·day⁻¹·mm⁻¹ Hg is equivalent to 945,600 ml (STP) \times mil/(m² \times day \times atm).

Waxes. Samples of commercial fruit waxes were obtained from American Machinery Corp., Orlando, Fla.; Brogdex Co., Pomona, Calif.; Bryler Creative Systems, Johnson Wax, Racine, Wis.; FMC Corp., Lakeland, Fla.; Fresh Mark Corp., Ocoee, Fla.; Inotek International Corp., Mentor, Ohio; and Pennwalt Corp., Decco Division, Monrovia, Calif. Wood rosin samples were from Brogdex Co. and Resinall, Hattiesburg, Miss., and carnauba wax emulsion was from American Machinery Corp. The shrink-wrap film was type D955, 60 gauge (0.015 mm) from Cryovac, Duncan, S.C. The formulations of the commercial waxes are proprietary, but available information is shown in Table 1. The 19 commercial wax coatings are samples A to S; the other materials are samples 1 to 5.

Permeability measurements. Permeability was measured by the method of Hagenmaier and Shaw (1991). The liquid coating was brushed onto plastic films of known high permeability; polyethylene-vinyl acetate film for O₂, CO₂, and C₂H₄ permeability; and cellulose acetate for water vapor. The films were cut into pieces to provide individual samples. The coating thickness for each sample was measured with a micrometer caliper and also calculated from the weight and density of the coating. A typical coating was 0.008 mm (0.3 mil) thick. Water vapor permeance of coated and uncoated samples was measured with

the Permatran-W1A water vapor permeability tester (Modern Controls, Minneapolis). Films were mounted with the cellulose acetate on the side with 0% relative humidity (RH) and the coating on the side with saturated NaCl (75% RH) or KNO₃ (92% RH).

Permeabilities of O₂, CO₂, and C₂H₄ were all measured on the same samples. Oxygen permeability was determined with the Ox-tran 100 permeability tester (Modern Controls) calibrated with standard reference material 1470, a polyester film from National Bureau of Standards. Carbon dioxide and C₂H₄ permeability were determined simultaneously, using gas made up of 50% each. Concentrations of these two gases were measured with a gas chromatography (Hewlett-Packard Model 5890A) fitted with a 30 m \times 0.53 mm id. polystyrene (GSQ) column (J&W Scientific, Folsom, Calif.) and a thermal conductivity detector. Column and detector temperature were 40 and 120C, respectively, and He carrier gas flow rate was 4.8 ml·min⁻¹. For standard reference material 1470, the measured CO₂ permeance was 11% below the certified value. For measurement of O₂, CO₂, and C₂H₄ permeabilities, RH was controlled to within five percentage points of the desired value by passing the incoming gases through bubblers kept at the appropriate wet bulb temperatures, and by using samples that had been conditioned beforehand in containers of controlled humidity. Coating permeability was calculated from the permeability of coated and uncoated films using Eqs. [1–3]. The reported permeability values, all measured at 30C, are mean values from three samples.

Results and Discussion

Permeability to O₂, CO₂, and C₂H₄ For the noncondensable gases, permeability to O₂, CO₂, and C₂H₄ is generally lower for the coatings with shellac and rosin than with carnauba and other waxes (Table 2). This observation fits the findings of

Table 1. Characteristics of 19 commercial wax coatings, a shrink-wrap film, and four related coatings.

Sample no.	Major ingredients	Recommended for
A	Waxes, natural and synthetic, and fatty acids	Muskmelon and stonefruit
B	Polyethylene and shellac	Lemon (for storage)
C	Carnauba wax and fatty acids	Peach, plum, nectarine
D	Carnauba wax and shellac	Pear, apple, citrus
E	Shellac, carnauba, fatty acids	Apple
F	Carnauba, fatty acids, shellac	Apple, pear, citrus
G	Coumarone-indene resin ^a	Citrus
H	Rosin and carnauba	Citrus
I	Hydrocarbon resins and fatty acids	Citrus
J	Rosin, oleic acid, and shellac	Citrus
K	Shellac and rosin	Citrus
L	Shellac and fatty acids	Citrus
M	Shellac and rosin	Citrus
N	Carnauba, shellac, and rosin	Citrus
O	Sucrose esters and carboxymethyl cellulose	Apples, avocado, banana, lime, melon, plantain, papaya, pear, pineapple, plum
P	Shellac and rosin	Citrus
Q	Shellac	Citrus
R	Shellac, rosin, and morpholine	Citrus
s	Shellac	Apple
1	Shrink-wrap film D955 (polyethylene-vinyl acetate copolymer)	
2	Carnauba wax, morpholine, and oleic acid	
3	Rosin, oleic acid, and NH ₃	
4	Shellac and, morpholine	
5	Modified maleic resin and morpholine	

^aSample G was the only solvent wax; all others were water-based.

Table 2. Permeability at 30C of 19 commercial wax coatings and five reference materials to O₂, CO₂, and C₂H₄.^a

Sample no.	CO ₂	C ₂ H ₄	O ₂	O ₂
	50% RH	50% RH	50% RH	85% RH
A	175,000 ^y	88,000 ^y	21,600 ^y	19,000
B	37,000	17,000	11,200	7,400 ^x
C	49,000	14,000	10,300	8,500
D	16,000	7,400	4,400	3,200 ^x
E	14,000	5,000	4,300	2,900 ^x
F	9,800	3,100	2,700	2,700
G	7,600	940	2,600	2,100
H	7,200	930	2,300	1,600 ^x
I	4,600	670	2,200	680 ^x
J	3,600	450	2,000	880 ^x
K	3,800	610	1,700	1,300
L	3,500	360	1,000	1,100
M	2,600	270	1,000	830
N	2,800	400	1,000	700 ^x
O	4,500	1,980	800	1,000
P	1,800	180	730	740
Q	1,900	170	640	770
R	1,700	140	550	440
S	1,700	180	470	750 ^x
1	27,000	12,000	9,100	8,900
2	7,800	3,200	2,000	1,500
3	2,200	170	780	1,100 ^x
4	1,100	90	370	430
5	910	310	250	490 ^x

^aPermeability expressed as ml (STP) × ml/(m² × day × atm).

^bCoefficient of variation is 10%.

^cOxygen permeability significantly different (*P* ≤ 0.05) at RH of 50% and 85%.

Ashley (1985), who notes that polymers containing hydroxy, ester, and other polar groups tend to have a lower O₂ permeability than polymers with hydrocarbon and other nonpolar groups. The O₂ content is 23% for shellac (Martin, 1982); » 8% to 20% for rosin, depending on the modification (Noller, 1965); 6% for carnauba wax (Bennet, 1975); and <5% for polyethylene wax (Food and Drug Administration, 1990).

Because of equipment limitations, the permeabilities were measured at 30C rather than at the temperatures used for refrigerated fruit storage. At lower temperatures the permeability is less, as has been shown by Rij and Mackey (1986) for plastic films. Shellac coatings at 0C have O₂ permeability an order of magnitude less than at 30C (Hagenmaier and Shaw, 1991).

RH also has an important effect on permeability. Some of the coatings have significantly different values of O₂ permeability at 50% and 85% RH (Student's *t* test, Table 2). This RH dependence is in large part due to the polar components used to raise pH and solubilize the polymer. For example, shellac solubilized with NaOH is much more permeable to O₂ than that solubilized with morpholine, especially above 85% RH (Hagenmaier and Shaw, 1991). Thus, at the high values of RH best suited for fruit storage, the permeability can be much higher than those given in Table 2.

Water vapor. Under the conditions of measurement, there was less variation in permeability to water vapor (Table 3) than to the other gases (Table 2). Still, the commercial waxes exhibit a 17-fold difference in water vapor permeability. As shown previously (Hagenmaier and Shaw, 1991) permeability to water vapor can be even more sensitive to RH than permeability to O₂, especially for a wax that contains polar ingredients.

Permeability of coated fruit. Consider the model where the

Table 3. Water vapor permeability at 30C of 19 commercial wax coatings and five reference materials at 75% or 92% RH on the coated side of the film.^a

Sample no.	75% RH	92% RH
	A	1.1
B	3.4	4.5 ^y
C	18	14
D	1.3	0.8
E	2.9	3.1
F	4.3	2.3 ^y
G	1.3	1.5
H	3.2	3.0
I	3.9	11 ^y
J	2.7	4.5 ^y
K	2.2	3.6 ^y
L	7.2	8.7 ^y
M	4.7	14.3 ^y
N	3.2	10 ^y
O	9.7	8.7
P	4.7	11 ^y
Q	5.0	5.8 ^y
R	5.3	12 ^y
S	9.1	9.6
1	0.20	0.22
2	0.8	0.6 ^y
3	1.7	30 ^y
4	4.4	19 ^y
5	2.5	3.6 ^y

^aPermeability expressed as g ml/m² · day⁻¹ · mm³ Hg. The mean coefficient of variation is 10%.

^bPermeabilities significantly different (*P* ≤ 0.05) at RH of 75% and 92%.

only pathway for gas exchange of an uncoated fruit is by active permeation through the fruit skin rather than by diffusion through open stomata. Now consider the same fruit with a coating. The peel and coating form a barrier equivalent to a laminate film. Thus, the permeance of the coated fruit relates to permeance of fruit and coating according to Eq. [3].

For example, an uncoated nonrefrigerated orange weighs 170 g, has 150 cm² of surface area, an internal partial O₂ pressure of 18%, and a respiration rate of 11 ml · kg⁻¹ · h⁻¹ (Ben-Yehoshua et al., 1985; Eaks and Ludi, 1960; Davis and Hofmann, 1973; Vines et al., 1968). From Eq. [1], the permeance of the peel is 100,000 ml (STP)/(m² × day × atm). Now consider the same fruit coated with a 0.05 -mil-thick coating (0.0013 mm) having an O₂ permeability of 2000 ml (STP) × ml/(m² × day × atm), a typical value for commercial coatings (Table 2). The permeance of the coating, from Eq. [2], is 40,000 ml (STP)/(m² × day × atm). The permeance of the coated fruit, from Eq. [3], is 29,000 ml (STP)/(m² × day × atm). Thus, permeance could be reduced from 90,000 to 28,000 ml (STP)/(m² × day × atm) by application of a coating to the fruit model. Alterations in gas exchange of the same magnitude have been observed from coating of nonrefrigerated real fruit (Banks, 1984a; Ben-Yehoshua et al., 1985; Meheriuk and Porritt, 1972; Trout et al., 1953; Vines et al., 1968). Thus, the permeation model described and the O₂ permeability data of coatings maybe useful in predicting the gas-exchange behavior of coated fruits and vegetables.

Relative permeability. Table 4 shows how permeability values are related to one another at 30C. As observed with polymers (Ashley, 1975), the ratios of noncondensable permeabilities

Table 4. Permeability of CO₂ and water vapor relative to that of O₂ for 19 commercial wax coatings and five reference materials.^a

Sample no.	CO ₂		Water vapor	
	Ratio ^b	SE ^c	Ratio ^b	SE ^c
A	8.1	2.1	38	4
B	3.3	0.5	380	22
C	4.8	0.5	1,300	230
D	3.6	0.3	160	12
E	3.2	0.7	660	34
F	3.6	0.5	800	130
G	2.9	0.4	550	50
H	3.1	0.3	1,200	90
I	2.0	0.5	4,500	1,400
J	1.9	0.5	2,200	470
K	2.3	0.3	2,100	200
L	3.4	0.5	8,000	700
M	2.5	0.2	13,000	2,800
N	2.7	0.2	9,300	1,500
O	5.6	0.5	10,000	1,200
P	2.4	0.8	14,000	2,600
Q	3.0	0.4	8,500	760
R	3.1	1.6	21,000	6,200
S	3.7	0.4	20,000	1,900
1	3.1	0.1	24	1
2	3.8	1.6	290	50
3	2.9	1.0	37,000	11,000
4	3.0	0.7	48,000	4,500
5	3.7	0.7	14,000	2,900

^aOxygen and CO₂ permeability at 50% RH, water vapor permeability at 92% RH on the coated side of the film.

^bThe CO₂ or water vapor permeability divided by O₂ permeability.

^cThe SE of the ratio was calculated from the variances and means of numerator and denominator.

vary much less than the permeabilities themselves. For fruit inside a continuous barrier of known permeability it follows from Eq. [1] that:

$$p^{CO_2}/p^{O_2} = (20.9 - O_2^i)/(CO_2^i - 0.03)/R.Q. \quad [4]$$

where R.Q. is the respiratory quotient (O₂ flux/CO₂ flux); 20.9 and 0.03 are ambient O₂ and CO₂ percentages, respectively, and PO₂ and PCO₂ are the O₂ and CO₂ permeances of the barrier.

A different treatment is appropriate when the main pathway for gas exchange is not by permeation but rather by diffusion through stomatal pores and the stem scar (Banks, 1984a, 1984b; Ben-Yehoshua et al., 1985; Bramlage, 1986; Burg, 1990; Clendenning, 1941; Meheriuk and Lau, 1988). In the case where the only pathway is by diffusion, the flux is proportional to the diffusion constant in air, which for CO₂ is 0.8 times that of O₂ (Burg, 1990; Nobel, 1974). Since 0.8 is much lower than any of the CO₂ ratios for fruit waxes (Table 2), calculation should permit some conclusions about the pathway for gas exchange in the coated fruit, which is related to the continuity of the coating. In the past, such analyses have been hampered by lack of information on permeability of the coating (Banks, 1984a; Ben-Yehoshua et al., 1985).

Ethylene. Ethylene permeability is important for diffusion of this gas into or out of the fruit. Unless it can permeate through the coating, it will accumulate inside the fruit and may adversely affect fruit quality, much as it does in a closed storage space (Knee, 1990; Watada, 1986). Diffusion of C₂H₄ into the fruit is important in degreening, but a wax barrier slows down the process (Jahn, 1976; Vakis, 1975). In both cases, high C₂H₄

permeability is desirable, which is best achieved with coatings made from waxes with little shellac or rosin (Tables 1 and 2).

Seal-wrapped fruit. Permeability measurements help to explain differences between seal-wrapped and coated fruit. The seal-wrap film had a very low permeability for water vapor and a relatively high one for O₂ and CO₂ compared to the fruit waxes (Tables 2 and 3). Seal-wrapped fruit has reduced transpiration and elevated respiration compared to fruit coated with coumarone-indene resin (Ben-Yehoshua et al., 1985; Hale et al., 1982; purvis, 1983).

Improvement of coatings. Wax coatings for citrus fruit differ markedly, thus some do not have the optimum permeabilities for the purpose intended. Permeability for citrus coatings should be high for O₂, CO₂, and C₂H₄ and low for water vapor to reduce transpiration as much as possible and not overly restrict respiration.

Permeability control may lead to a general improvement in the technology of fruit coatings. First, data are needed on the performance of coatings of known permeability and thickness. Much of the work done on performance of coatings cannot be used: commercial coating formulations change often, and therefore a coating's name is not sufficient identity. Moreover, thickness of coating has almost never been reported. Second, information is needed on what coating permeance is optimum for storage of various commodities. Finally, coatings need to be manufactured and used according to the permeability specifications. A similar approach was used for the plastic film that forms a permeability barrier for seal-wrapped fruit and vegetables (Daun et al., 1973; Gates, 1988).

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