# Moisture Permeability of Edible Films Made with Fatty Acid and (Hydroxypropyl)methylcellulose

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Edible films made up of 55% (hydroxypropyl)methylcellulose and 45% stearic acid had permeability of  $0.17 \pm 0.04$  g mil/(m<sup>2</sup> day mmHg), evaluated with 85% relative humidity on one side of the film, 0% on the other. The permeability was unchanged at 94% relative humidity. It was higher at 97– 99% relative humidity and also higher if shorter chain fatty acids were substituted for stearic acid. Permeability increased 10-fold when stearic acid crystals were removed from the surfaces of the film. The permeability was higher when the more crystalline side was toward the high-humidity atmosphere. Tensile strength of the film decreased rapidly at values of relative humidity above 90% on both sides of the film.

# INTRODUCTION

Edible films are thin protective coatings that are intended to be eaten. Protective coatings have a long history of usage on foods (Guilbert, 1986). The usage of edible films is predicted to increase (Sills-Levy, 1989).

Edible films are sometimes used to form a boundary between two components within the same food, whereas plastic packaging films generally form a boundary between the food and the outside world. An example of an edible coating to control moisture content within a food is a coating used in a breakfast cereal to retard the movement of water from the raisins into the cereal flakes, thus causing loss of crispness.

Recent work at the University of Wisconsin has resulted in the development of some edible films with good barrier properties (Kamper and Fennema, 1984a,b, 1985; Kester, 1988; Kester and Fennema, 1986, 1989a,b; Greener and Fennema, 1989a,b). Of particular relevance to this work were films made from cellulose derivatives and fatty acids. The present work further explores the limits and properties of these films and is restricted to development of films to regulate moisture transfer. It is further limited to cast films rather than those films formed directly on the surface of a food product.

To help assure the edibility of the films, the ingredients used in this study were limited to those approved for use in foods under 21 CFR 172, 182, or 184 (CFR, 1988a-c). Furthermore, the ingredients are approved without numerical limit, the only restriction being good manufacturing practice.

# MATERIALS AND METHODS

The (hydroxypropyl)methylcellulose (HPMC) was Methocel E15LV Premium Grade from Dow Chemical Co., with 12% hydroxypropyl and 30% methoxy substitution. The weight-average molecular weight was approximately 50 000. The fatty acids were 99% pure chemicals from Sigma Chemical Co. and also USP stearic acid from Fisher Scientific. Linear low density polyethylene (LLDPE) was supplied by Dow. The rated water vapor transmission rate (WVTR) for 2-mil LLDPE (called Tybrite 126) is 0.73 g/(m<sup>2</sup> day) at 90% relative humidity, 100 °F (Dow, 1987).

**Casting of Film.** To make films, the E15LV was dispersed as a 1-4% suspension in 95.6% ethanol at 5–10 °C. Fatty acid was added; the solution was heated to 50 °C, filtered through glass wool, vacuum deaerated, and dried on the same day. To cast and dry the film, 20 g of the solution was poured into a hot, polished stainless steel pan measuring 20 cm i.d. at the bottom. While drying, this pan was rotated at 0.5 rpm to help compensate for any deviation from absolute levelness of the drying surface. Drying was at 75 °C air temperature for 1 h. The temperature of the alcohol solution was maintained at about 60 °C by controlling the evaporation rate at about 1.3 mg/(min cm<sup>2</sup>). To effect this control, a cover was used that had openings of 0.6% of the film area. Thus, the solution was kept above the temperature at which stearic acid precipitates from 95% ethanol, namely, 37 °C for 10%, 42 °C for 20%, and 46 °C for 30% stearic acid (Ralston, 1948).

After the films were dried, they were, with one exception, cooled immediately at ambient temperature (about 25 °C). The films were peeled off while the pan was still slightly warm. For the exception noted, the air temperature after drying was lowered to 60 °C, and the films were tempered there overnight.

The cast films were stored at  $25 \, ^{\circ}$ C, 50% relative humidity, for at least 1 day before being used for any measurements. During this first day the films containing fatty acids shrank somewhat, and they also developed a surface layer of fatty acid crystals, a phenomenon previously noted by Kester and Fennema (1989a).

**Film Properties.** Direct measures of thickness were made to 0.001-mm reading with a micrometer having 6 mm diameter measuring faces. Surface density was measured by weighing to 0.1 mg a section of film with area approximately 30 cm<sup>2</sup>.

Film tensile properties were measured by puncturing, at 20 cm/min with a round-ended rod of 8-mm diameter, a piece of film clamped over a 2.5 cm diameter hole. For calculation of tensile properties, the cross-sectional area was taken as thickness of the film times circumference of the 2.5-cm hole. Puncture strength was calculated as the peak load per area. Energy to puncture was calculated as the integral of load per area times plunger distance to break point. Measurements were made at ambient conditions (50% relative humidity, 25 °C), the same conditions used for storage of the film before testing. The instrument used was an Instron Model 1011.

Water Vapor Transmission Measurements. Water vapor transmission rate was measured at 27 °C by the traditional cup method (ASTM, 1987). Per section 11 of that method the relative humidity (RH) was maintained on the low-humidity side of the film with  $CaCl_2$  suspended on nylon cloth about 6-mm distance above the film. Per section 12, the relative humidity on the high-humidity side was controlled by filling the cup to within 6 mm of the film with a solution of known water activity. For example, 4.0 molal NaCl was used for 85% relative humidity (Washburn, 1928). The lid and cup, both made of 9 cm Petri dish bottoms, were stacked one on the other and clamped in position.

Film samples were sealed with beeswax onto heavy-duty (0.025 mm) aluminum foil. The film, in effect, was a patch over

 Table I.
 Puncture Test of Films Containing Different

 Fatty Acids<sup>4</sup>

fatty acid <sup>b</sup>	fatty acid <sup>b</sup> strength, <sup>c</sup> lb/in <sup>2</sup>	
stearic, C <sub>18</sub>	$651 \pm 60$	$5.1 \pm 0.8$
palmitic, C <sub>16</sub>	686 ± 95	$6.0 \pm 1.2$
myristic, C <sub>14</sub>	763 ± 95	$8.2 \pm 1.2$
lauric, C <sub>12</sub>	$608 \pm 95$	$6.8 \pm 1.2$

<sup>a</sup> The films contain 45% fatty acid and weigh  $2.0 \pm 0.2 \text{ mg/cm}^2$ . Measurements are at 50% relative humidity. <sup>b</sup> The results are from 20 trials with stearic acid and 8 with the other fatty acids. <sup>c</sup> The ranges show 95% confidence.

a measured opening  $(2-30 \text{ cm}^2)$  cut into the foil. The foil was, in turn, sealed to the cup with 45 °C melting point paraffin. Assembled cells were kept at 27 °C; the cup was weighed one to three times daily to an accuracy of 0.1 mg. The permeability rate was determined from least-squares analysis of weight versus time after steady-state conditions were achieved, about 1 day after the film was installed in the permeability cells.

### **RESULTS AND DISCUSSION**

**Thickness.** The uniformity of film thickness is of some importance because other properties in turn depend on it. The thickness of 19 samples of HPMC-stearic acid film was measured with a micrometer at four places about 2 cm from the edge of the film and also twice near the center. The mean standard deviation within the films was 0.13 mil, about 10% of the average thickness. The centers were  $0.23 \pm 0.05$  mil thinner than the edges, which is attributed to the fact that the drying surface was slightly elevated in the center.

Micrometer values of film thickness were influenced by the roughness of the film. The micrometer thickness was  $0.52 \pm 0.05$  mil higher than that obtained from measured weight per unit area divided by known values of specific gravity [1.39 for E15LV films (Dow, 1989); 0.94 for stearic acid (Weast, 1976)]. An explanation for the high micrometer readings is that the elevated spots on the films (evidently the larger crystals) stopped the micrometer faces short of the surface. The values of thickness determined from film weight are considered more reliable, and so these were the ones used in calculation of permeability and tensile properties.

**Film Integrity.** Tensile strength is of importance for two reasons. First, it was not possible to measure moisture permeability if the film fell apart during the experiment. Second, successful use of a film that is formed on the surface of a food seems to require that the film have some degree of structural integrity. Preliminary trials with coatings that were not films showed these to be ineffective barriers.

Table I shows that the puncture properties of HPMCfatty acid film are not influenced by the chain length of the fatty acid. All the films in Table I had sufficient toughness for measurement of permeability. The minimum energy requirement for that purpose seemed to be about 0.5 ft lb/in<sup>2</sup>. The values in Table I were compared with 1-mil LLDPE, measured by the same test. For LLDPE the puncture strength was 765  $\pm$  30 lb/in<sup>2</sup>, and the puncture energy was 33  $\pm$  4 ft lb/in<sup>2</sup>. Relative to polyethylene the HPMC-fatty acid films have similar strength but require only about 20% as much energy to puncture. The reason for this is that polyethylene is much more elastic than the HPMC-fatty acid films.

The physical properties of the film depend somewhat on the fatty acid component. First, a small amount of fatty acid, about 2%, was needed to enable the films to be readily peeled from the drying surface. Second, as Figure 1 shows, the energy to puncture increased as stearic acid replaced



Figure 1. Energy to puncture at different levels of stearic acid. Each data point represents at least eight trials, all measured at  $50 \pm 5\%$  relative humidity. The error bars show 95% confidence ranges.



Figure 2. Energy to puncture at different levels of relative humidity. Stearic acid content is 42%. Energy is the percentage of its value for the same film at 50% relative humidity. Each data point represents at least two trials.

the polymer, reaching a maximum at about 30% stearic acid. Although not used to make the film tougher, the stearic acid had that side effect. Further, at stearic acid content above 70% the film was not homogeneous. Finally, with stearic acid content at 100% no film was formed. In summary, cast films could be made with 2–65% stearic acid.

Film strength is also dependent on relative humidity. Pieces of HPMC-stearic acid film were equilibrated against air of controlled relative humidity. The puncture properties were then measured at 50% relative humidity within 2 min after the film was removed from the controlled humidity environment. The results, given in Figure 2, show that puncture energy deteriorated to a very low value at relative humidity of about 97% on both sides of the film. However, as will be shown later, these films still have value as barriers with relative humidity as high as 97% on just one side of the film. The data in Figure 2 are for films with 42% stearic acid. Films with 15% or 60% stearic acid also became very weak at relative humidity above 97%.

**Permeability.** The effectiveness of various films in retarding moisture loss was evaluated from values of water vapor transmission rate, measured as the weight of water (g) per unit area (m<sup>2</sup>) and time (day). For most gases, WVTR is proportional to  $\Delta p$  and inversely proportional to L, where  $\Delta p$  is the difference in the partial pressure (mmHg) of a gas across the film and L is the thickness of the film in mils (0.001 in.). The proportionality constant is the permeability (P), which has units of g mil/(m<sup>2</sup> day mmHg), hence, the well-known equation

#### WVTR = $P(\Delta p/L)$

P is often not a constant when the diffusing substance is water vapor, especially when the film is an edible one.

Table II. Permeability of HPMC Films with No Lipids Added

film wt, mg/cm <sup>2</sup>	film thickness, mil	permeability,ª g/m² day mmHg	
4.9	1.5	60	
3.5	1.1	51	
2.3	0.70	32	
1.0	0.31	22	

<sup>a</sup> Each P value is the average of two observations at 85% relative humidity. Differences in P of 21 g mil/(m<sup>2</sup> day mmHg) are significant at the 95% level.



**Figure 3.** Permeability  $[g \min!/(m^2 \operatorname{day} mmHg)]$  as a function of fatty acid chain length for films weighing 1.8–2.3 mg/cm<sup>2</sup>. Each data point represents at least eight trials at 85% relative humidity. 27 °C. (**A**) 45% fatty acid; ( $\nabla$ ) 23% fatty acid.

The reason is that the structure and properties of edible films (like most foods) are strongly affected by relative humidity. Thus, the values of P reported here and elsewhere apply only to the particular vapor pressures used for measurement.

Table II shows the permeability of HPMC films with no lipid added. The values of P are high, similar to the permeabilities of other lipid-free edible films (Schultz et al., 1949; Rankin et al., 1958).

Film permeability is very dependent on type and quantity of fatty acid. Figure 3 shows that the longer chain fatty acids make better barriers, in agreement with the findings of others (Kamper and Fennema, 1984a; Jarvis et al., 1962). The film with 45% stearic acid had the lowest permeability, amounting to 0.14 g mil/( $m^2$  day mmHg). The film with 45% palmitic acid also had fairly low permeability:  $0.3 \text{ g mil}/(\text{m}^2 \text{ day mmHg})$ . However, permeability of the palmitic acid film increased 30-fold when the fatty acid content was lowered from 45% to 23%. Because the permeability is so strongly dependent on the amount of palmitic acid, good control of film permeability may be difficult with that fatty acid, and therefore stearic acid is the lipid of choice. Since the longest chain fatty acid gave the best barrier, it would seem promising to try fatty acids with chain length longer than stearic. However, the FDA regulation covering fatty acids (21 CFR 172, 1988a) does not specifically allow such fatty acids as food additives.

The data in Table III show that stearic acid content of about 40–50% gives lowest permeability. Films with that composition not only have the lowest but also the least variation in permeability. With less stearic acid the permeability was higher, sharply dependent on stearic acid content, and also quite variable at any given film composition. The data in Figure 3 and Table III, taken together, show the difficulty in making judgment on the value of a lipid when the composition of film is not optimum for low permeability. For example, palmitic acid (C<sub>16</sub>) might have been judged almost equivalent to stearic acid (C<sub>18</sub>) had they been compared only at the 45% level. Stearic acid itself might have been rejected had it been evaluated at

Table III. Permeability of Stearic Acid-HPMC Films\*

stearic acid, <sup>b</sup> %	permeability, g mil/(m² day mmHg)
0	48 € 18°
2	31 ± 9
14	$2.1 \pm 1.8$
24	$0.5 \pm 0.4$
40	0.16  0.07
45	$0.14 \pm 0.04$
56	$0.23 \pm 0.09$

<sup>a</sup> Permeability values are based on four trials at 85% relative humidity and 27 °C. <sup>b</sup> The films weighed  $2.4 \pm 0.4 \text{ mg/cm}^2$  and had thickness of  $0.75 \pm 0.1 \text{ mil.}$  <sup>c</sup> The confidence ranges show 95% significance.

Table IV.	Permeability	of Films	Containing	42 ±	: 3%
Stearic Aci	ide 🦷				

film wt, mg/cm <sup>2</sup>	thickness, mil	permeability, g mil/(m² day mmHg)	
0.46	0.15	$3.5 \pm 1.4$	
0.94	0.32	$0.29 \pm 0.20$	
1.70	0.58	$0.20 \pm 0.10$	
2.07	0.72	$0.14 \pm 0.04$	
4.25	1.46	$0.20 \pm 0.11$	

 $^a$  Each P value is for at least four trials. The range is for 95 % confidence.

the 14% level, where permeability was high, or at 75% stearic acid, where the film was not homogeneous. Each lipid must be evaluated under its own optimum range of conditions.

Table IV shows the permeability of films at different thicknesses, but the same composition. Permeability was independent of thickness for 0.6-1.5-mil films. The average P in that range is  $0.17 \pm 0.04$  g mil/(m<sup>2</sup> day mmHg), on the basis of 16 trials. This compares to our measured value for LLDPE of  $0.24 \pm 0.05$  (10 trials). The HPMC-stearic acid film has lower permeability than LLDPE, and the difference is significant at the 95% level. Note that the manufacturer's value for WVTR of LLDPE, cited earlier, is equivalent 0.33 g mil/(m<sup>2</sup> day mmHg).

Table IV also shows that the permeability is high for thin films of HPMC-stearic acid. At 0.15-mil thickness  $(0.46 \text{ mg/cm}^2)$ , the permeability was about 20 times its value for films of 0.6-1.5-mil thickness. One possible explanation is that the very thin films stretch more when peeled from the drying surface. Other results, not shown here, indicate that the HPMC-stearic acid films have much higher permeability when the films have been stretched slightly. Results from Tables III and IV together show that films with stearic acid below about 0.5 mg/cm<sup>2</sup> do not make good barriers, a conclusion also in line with data from Kamper and Fennema (1984b).

The high permeability at low levels of stearic acid is a significant disadvantage should this film be formed on a food surface where film thickness is poorly controlled or in cases where the film becomes diluted with other food components. For example, if 20% of the surface were covered by a film having permeability of 5 g mil/(m<sup>2</sup> mmHg day), then the film as a whole would have P of at least 1.0 g mil/(m<sup>2</sup> mmHg day), about 6 times as high as a good HPMC-stearic acid film. Thus, for good protection of a food, the uniformity of the coating is more important than any further reduction of P below the already achieved value of 0.17 g mil/(m<sup>2</sup> mmHg day).

**Factors Increasing Permeability.** How the film is handled is also important. As already noted, HPMCstearic acid films became covered with stearic acid crystals. The following experiment tested the importance to permeability of this surface layer of fatty acid. Films were

Table V. Film before and after Removing Surface Crystals<sup>a</sup>

	film wt, mg/cm <sup>2</sup>	permeability, g mil/(m² day mmHg)
before	1.71	$0.19 \pm 0.17$
after	1.64	2.4 ± 0.4

 $^a$  Average values for three trials with a film containing 40% stearic acid, 0.57 mil thick.

Table VI. Permeability at Different Film Orientations

% R <b>H</b> ⁴	no. of trials	ratio <sup>b</sup>
85	5	$0.54 \pm 0.47^{\circ}$
97	3	$0.66 \pm 0.44$
combined	8	$0.58 \pm 0.40$

<sup>a</sup> The film used at 85% relative humidity had 40% stearic acid; that used at 97% relative humidity had 61% stearic acid. <sup>b</sup> Each film was tested at both orientations. The ratio is P (rough side toward desiccant) divided by P (film reversed). <sup>c</sup> The range shows 95% significance.

made of 40% stearic acid and 60% HPMC. During the first day after preparation the films shrank by 7% in length (average values for eight films). On day 5 the surface powder was wiped off with tissue paper, resulting in films that were fairly transparent.

Viewed at  $4.5 \times$  magnification, the film with crystals wiped off looked clear except for some white coating in cracks and crevices on the film. By contrast, the control film looked somewhat different. The rough (air-interface) surface appeared to consist of random piles of shiny white granules. The smooth surface was dull white except for craters presumably formed by out-migration of fatty acid.

The films were weighed before and after the surface powder was removed. Film weight and permeability (85% relative humidity) were measured for three samples of the original and wiped film. Table V shows that for this film more than a 10-fold increase in permeability resulted from wiping off the surface crystals, even though these amounted to only 0.07 mg/cm<sup>2</sup> or 4% of the weight of the film. A possible explanation is that the small amount of stearic acid on the surfaces was more important in blocking water vapor transmission than was the bulk of the stearic acid trapped in the polymer matrix. This hypothesis, however, does not explain why very thin films had much higher permeability. Both cases of high permeability could possibly have been caused by pinholes, since these were not routinely tested for.

As described under Materials and Methods, some films were tempered overnight at 60 °C, which is 7 °C above the  $\alpha$ - $\beta$  crystal transition temperature for stearic acid (Ralston, 1948). These particular films contained 40% stearic acid and weighed 1.7 mg/cm<sup>2</sup>. The tempered film had P of 0.30 mil g/(m<sup>2</sup> mmHg), compared with 0.19 for the untempered film. Although that difference was significant at only the 80% level, it does indicate that no improvement in permeability resulted from tempering the film. Kester (1988) also found only slight differences in water vapor permeability for different crystalline forms of fatty acid.

Permeability was measured with different orientations of the film. The data of Table VI show that the permeability is significantly higher, at the 95% level, when the rougher, more crystalline film surface is on the high humidity side of the film. A possible explanation comes from the observation that transmission rates sometimes vary more with the pressure at the ingoing surface than the outgoing surface, suggesting that the high-humidity surface is more critical (Lebovits, 1966). A surface that is rough would seem to have a higher effective surface area.

Table VII. Permeability at Different Levels of Relative Humidity<sup>4</sup> (RH)

		P, g mil/(n	n² day mmHg	)
stearic acid, %	99% RH	97% RH	94% RH	85% RH
61 57 52 mean	$2.1 \pm 0.7$ $2.6 \pm 1.6$ $2.2 \pm 1.3$ $2.3 \pm 0.6$	$0.84 \pm 0.4$ $0.57 \pm 0.1$ $1.8 \pm 1.1$ $1.1 \pm 0.5$	$0.30 \pm 0.13$ $0.38 \pm 0.18$ $0.08 \pm 0.03$ $0.25 \pm 0.11$	$\begin{array}{c} 0.16 \pm 0.07 \\ 0.23 \pm 0.13 \\ 0.28 \pm 0.12 \\ 0.22 \pm 0.07 \end{array}$

<sup>a</sup> All films had 0.9 mg/cm<sup>2</sup> HPMC. One side of the film was at the specified relative humidity, the other side at 0% relative humidity. Each *P* value is the average of three trials.

When this larger effective area is on the critical side of the film, the permeability is higher.

The permeability of films at high levels of relative humidity is shown in Table VII. Again, these relative humidity values relate only to the air on one side of the film. The mean values indicate that as relative humidity was raised from 85% to 94% there was no change in permeability. At 97% relative humidity the average permeability was about 4 times its value at 94% relative humidity, and at 99% relative humidity it again doubled. The mean permeability at  $97\,\%\,$  relative humidity is  $60\,\%\,$ of the value observed by Kamper and Fennema (1984b) from a blend of stearic and palmitic acids. Thus, it may be possible to use the HPMC-stearic acid films at high humidity without need for the beeswax coating using by Kester (1988) for that purpose. The very low limit set on beeswax in food, namely 0.002% (21 CFR, 1988c), makes it almost impossible to use this ingredient in edible films.

In summary, films made with (hydroxypropyl)methylcellulose have low values of water vapor permeability providing, first, they not be used at relative humidity values above 97%; second, the stearic acid exceed  $0.5 \text{ mg/cm}^2$  of film surface; third, the less crystalline side of the film face the higher humidity; and fourth, the films not be subjected to abrasion.

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**Registry No.** Hydroxypropyl methyl cellulose, 9004-65-3; stearic acid, 57-11-4; palmitic acid, 57-10-3; myristic acid, 544-63-8; lauric acid, 143-07-7; water, 7732-18-5.