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MECHANICAL AND BARRIER PROPERTIES OF EDIBLE CORN AND WHEAT PROTEIN FILMS

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ABSTRACT

Published methods for production of homogeneous edible films from corn and wheat proteins were adapted. Barrier and mechanical properties of the edible films were evaluated with procedures commonly used on polymeric films. Mechanical property data included thickness, elongation, tensile strength, tear strength, and burst strength measurements. Barrier property data included water vapor, oxygen, and carbon dioxide gas transmission rate measurements. Homogeneous corn and wheat protein films were found to have low tensile strengths, far less than cellophane. Corn films were brittle while wheat films were elastic in comparison to cellophane. All three types of film had low permeabilities for dry gases but relatively high water vapor permeabilities. **KEYWORDS.** Corn, Wheat, Edible films, Barrier properties, Mechanical properties, Physical properties.

INTRODUCTION

Deterioration of fresh foods has long been a problem for consumers. Development of methods to slow or stop deterioration is a popular topic among food processors. A multitude of food processing equipment and materials have been designed for this task. Included are polymeric films which slow the desiccation of product, provide a barrier to many other harmful environmental influences, and act as protection from external impinging energy.

The idea of using edible materials as a protective coating is not a new one. Indeed some fruits and vegetables come with their own natural protective coating like the skin on peaches and pears. If an edible film could be developed which reduced harmful effects to the environment such as desiccation or bruising, the consumer and producer would benefit from extended shelf-life and higher quality products. Advantages of using edible materials include being able to consume the film with the product and reducing the use of plastics or other packaging, which has to be discarded after use.

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Currently, many edible films exist on a research scale. Very few have been developed and used commercially. Edible films are usually categorized into three categories; polysaccharide, lipid, and protein films (Kester and Fennema, 1986). A corn protein film was developed by Osborne (1897). Anker et al. (1972) developed a film based on wheat gluten.

Presently, very little is known about the barrier and physical characteristics of most edible films. Commercialization of such films would be enhanced if the films were better characterized. This study defined some of these unknown characteristics for edible films produced in the laboratory. This study also determined if the methods of production consistently produced homogeneous films.

OBJECTIVES

The objectives of this study were to:

- Adapt a method for consistently forming homogeneous films made of corn and wheat proteins, respectively.
- Determine physical and permeability characteristics for the protein films.
- Compare characteristics observed for edible protein films with those of a commercial film, cellophane.

PROCEDURES

The films produced in this study were derived from corn and wheat proteins. Added constituents were kept to a minimum to reflect the properties of pure protein films as much as possible. Methods of production were modified literature methods developed during previous studies (Aydt and Weller, 1988; Aydt et al., 1989).

American Society of Testing and Materials (ASTM, 1987) standard tests were used to determine the properties of the edible films produced. Testing methods approved for plastics were used since a comparison was desired between edible and polymeric films.

CORN PROTEIN FILM PRODUCTION

An aluminum mold with cross-sectional dimensions of 11.4 × 43.1 cm and a well, 0.061-cm deep, was used in casting corn protein films (fig. 1). The commercial product, Cozeen® 303NF* produced by Zumbro Inc. (Hayfield, MN) served as the source of corn protein. This product

*Trade names are used in this publication solely for the purpose of providing specific information. Mention of a trade name, proprietary product, specific equipment or company does not constitute a guarantee or warranty from Clemson University and does not imply approval of the named product to the exclusion of other products that may also be suitable.

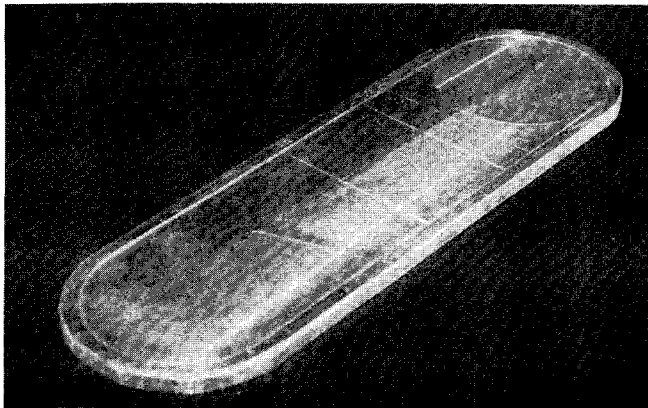


Figure 1—Mold for casting corn protein films.

contains the protein fraction, zein, solubilized in an ethanol and glycerin solution. The zein-ethanol solution was poured into the well and a straight edge passed along the top edges of the well to remove excess material. Films formed as the ethanol evaporated. Film drying was done in a hood at $23 \pm 1^\circ \text{C}$ for 24 h. After drying, films were cut to specific dimensions for testing and then peeled from the well.

WHEAT PROTEIN FILM PRODUCTION

The method of Anker et al. (1972) was adapted for use in this study to produce wheat protein films. With the method, 15 g of wheat gluten were added to 72 mL of 95% ethanol and 6 g of glycerol. A wheat gluten fraction, DO-PEP®, obtained from ADM Arkady (Olathe, KS), was used. This product contained about 75% protein, dry solids and a maximum of 7.5% moisture. The gluten solution was then warmed and stirred while 48 mL of water and 12 mL of 6 N $\text{NH}_4 \text{OH}$ were added. The solution was further heated to boiling to disperse the wheat gluten. Dispersion was indicated by the solution becoming less viscous. The dispersed mixture was cast onto a clean flat non-reactive surface (plate glass) using a thin-layer chromatography spreader bar produced by Brinkman Co. (New York, NY) set to a 0.2 cm gap. Film drying was done in a hood at $23 \pm 1^\circ \text{C}$. After 24 h of drying, each film was cut to specific dimensions for testing and slowly peeled from the plate glass to prevent distortion.

CONDITIONING OF MATERIALS

Physical and gas transmission properties of most films are influenced by temperature and relative humidity. These conditions must be standardized before a reliable comparison can be made of different materials. It was necessary to subject the materials to controlled environmental conditions prior to and, when possible, during testing. Films were conditioned at 50% relative humidity (RH) and 26°C for 72 h before physical tests were performed as described in the ASTM Standard Method D 618-61 (ASTM, 1987). A temperature of 26°C was used instead of the suggested 23°C because the room used for testing was maintained at this temperature. RH of 50% was obtained by using a saturated calcium nitrate solution in a desiccator.

Film conditioning before gas transmission tests required subjecting films to 0% RH at 26°C for 72 h. These

conditions were not maintained during gas transmission testing since other conditions were called for during other testing as noted in the sections describing these other tests. RH of 0% was obtained using calcium sulfate in a desiccator.

THICKNESS

Thickness was measured using an Ames thickness measuring device (B. Ames Co., Waltham, MA). Measurements were to the nearest $13 \mu\text{m}$ (0.5 mil). Five measurements were randomly taken on each specific test cut of film and a mean thickness was calculated for each film.

TENSILE AND ELONGATION

Three specimens strips, $6.00 \text{ cm} \times 2.54 \text{ cm}$ (2.36 in. \times 1 in.), were cut from each sample film produced. They were then peeled from the glass surface, conditioned, and measured for thickness. An Instron Universal Testing Instrument (Model No. 5011 7012 11, Instron Engineering Corp., Canton, MA) was used to measure the amount of uniaxial force necessary to pull each strip apart or its tensile strength according to ASTM Standard Method D 882-83 (ASTM, 1987). Film strips were placed in 483-kPa (70-psi) air-actuated grips on the universal testing machine. Initial grip separation was 4.0 cm (1.6 in.) with a crosshead speed of 3.0 cm/min (1.2 in./min). Chart speed was set at 50.8 cm/min (20 in./min). Percentage elongation at break was calculated, according to the above ASTM Standard Method, by dividing the elongation at the moment of rupture of the specimen by the initial length of the specimen and multiplying by 100. Tensile strength is an important characteristic of films with a potential for heavy-duty use and elongation gives a good indication of toughness. A large elongation indicates a film's ability to absorb a large amount of energy before breaking.

TEAR PROPAGATION

An Elmendorf Tear Tester (TMI Co., Amityville, NY) was used to determine the force necessary to continue tearing a film specimen after it had initially been cut. Following the procedures described in ASTM Standard Method D 1922-67 (ASTM, 1987), two film sections of $76 \text{ mm} \times 63 \text{ mm}$ were cut with a sample cutter from each test film produced and their thickness measured. The 76 mm length of each section was used as the direction of tear. A 20 mm slit was made at the center of the edge perpendicular to the direction being tested, making the test length 56 mm. The capacity of the pendulum used on the Elmendorf Tear Tester was 12.3 g.

BURSTING STRENGTH

One film section of $7.6 \text{ cm} \times 7.6 \text{ cm}$ (3 in. \times 3 in.) was cut from each of the test films produced, peeled from the glass, and conditioned. Testing followed the procedures of ASTM Standard Method D 774-67 (ASTM, 1987) using a Mullen Burst Tester (TMI Co., Amityville, NY). Specimens were placed in the clamps of the tester with the smooth side of the film facing the diaphragm. A bourdon gauge measured the hydrostatic pressure needed to burst the film. Bursting strength was determined only for wheat films because corn films were too brittle to mount properly in the tester. Corn films continually broke under stress of

the clamping device used to hold films in place in the tester.

WATER VAPOR TRANSMISSION

ASTM Standard Method E 96-80 (ASTM, 1987) was used to determine water vapor transmission by sealing a known open area of an impermeable container with a film. Covered aluminum moisture dishes of 9.5 cm diameter and 5 cm deep, were filled with distilled water to a level of 1 cm from the top so as to impart a RH of 100% beneath the films. Films were sealed with clear silicon onto the dish lids over circular holes of 3.2 cm (1.25 in.) in diameter. The covered containers were periodically weighed while being held in an environmental chamber at 26° C. RH was maintained in the chamber at 50% with a saturated calcium nitrate solution. A linear regression analysis was performed with weight loss as the dependent variable and elapsed time as the independent variable. The slope of the regression curve divided by the area of the opening was defined as the water vapor transmission (WVT). WVT divided by the vapor pressure difference across the film is considered the permeance of the film. Permeance multiplied by the thickness of the film is known as the permeability.

OXYGEN GAS TRANSMISSION

A 100 cm² (15.5 in.²) diffusion cell, incorporated in a MOCON Oxtran 100 (Modern Controls, Inc., Minneapolis, MN), was used to measure O₂ gas permeability using a coulometric sensor. The MOCON equipment provides a millivolt voltage signal with a sensitivity of 1 mV per output of 100 cm³/cm² · 24 h (0.155 cm³ / 100 in.² · 24 h). The procedure followed ASTM Standard Method D 3985 - 81 (ASTM, 1987) and is referred to as an isostatic method for measuring permeability of a barrier film (Giacin et al., 1984).

After conditioning, test films were placed in the cell under conditions of 37.8° C (100° F) and 0% RH. Pure (100%) O₂ was allowed to pass by one side of the films. Nitrogen, a carrier gas flowing on the other side of the film, carried transmitted O₂ to the coulometric detector for measurement. Permeability was calculated by multiplying the rate of O₂ gas transmission by the thickness of the film and then dividing by the partial pressure.

Corn protein films cracked when clamped into the cell because the films were too brittle. Therefore, a self-adhesive aluminum foil mask was used to reduce the area of each film to 10 cm² (1.55 in.²). More importantly the foil mask added strength to the films on the surfaces clamped into the cell. Corn protein films were masked before conditioning.

CARBON DIOXIDE GAS TRANSMISSION

A three-chamber Gilbert and Pegaz gas cell was used to measure CO₂ gas permeability following a procedure described by Gilbert and Pegaz (1969). This procedure, a quasi-isostatic method for measuring CO₂ gas transmission rate of a barrier film (Giacin et al., 1984), was conducted at 22.8° C (73° F) and 0% RH. In the method, 100% CO₂ was passed through the two outer chambers of the cell at a constant flow rate at a pressure of 1 atmosphere (atm). Carbon dioxide permeated through two specimen films and was collected in the center chamber of the cell. The center

chamber had been previously flushed with nitrogen, an inert gas, and closed off from external air.

Carbon dioxide was not allowed to exceed 1 to 2% in the center chamber (volume of 75 cm³) during testing, thus maintaining a constant driving force. At predetermined intervals, aliquots were withdrawn from the center chamber and analyzed on a gas chromatograph. The gas chromatograph was equipped with a 100/120 Carbosieve S-II 3 m × 0.3 cm (10 ft × 0.125 in.) stainless steel column and its thermal conductivity detector had a HWD current setting of 225 mA. Manifold, column, and injection temperatures were 250, 200, and 200° C, respectively. Sample size for injection was 0.2 μL.

Concentration of CO₂ was plotted as a function of run time. Transmission rate was obtained from the slope of the linear portion of the time profile curve (see example in fig. 2) multiplied by the chamber volume and then divided by the test area of the film; 10 cm² (1.55 in.²) for corn protein films and 50 cm² (17.75 in.²) for wheat protein films. Permeability was determined by multiplying the transmission rate by the thickness of the film and then dividing by the partial pressure.

Film specimens used in the CO₂ transmission tests were cut from the same cast film as specimens for O₂ testing. Each specimen had a 100 cm² (15.5 in.²) circular area. Two specimens were cut from each cast film sample and conditioned.

DATA ANALYSIS

Six or more replications of each test were performed. The actual number of repetitions varied for each test and is shown in Table 1. Mean, range, and coefficient of variation (CV) were determined for each test. The mean values of the edible films were compared to values for a commercial film, cellophane. Observed values for certain characteristics of cellophane were collated with other values from a specification sheet for the film produced by Flexel, Inc. (Covington, IN).

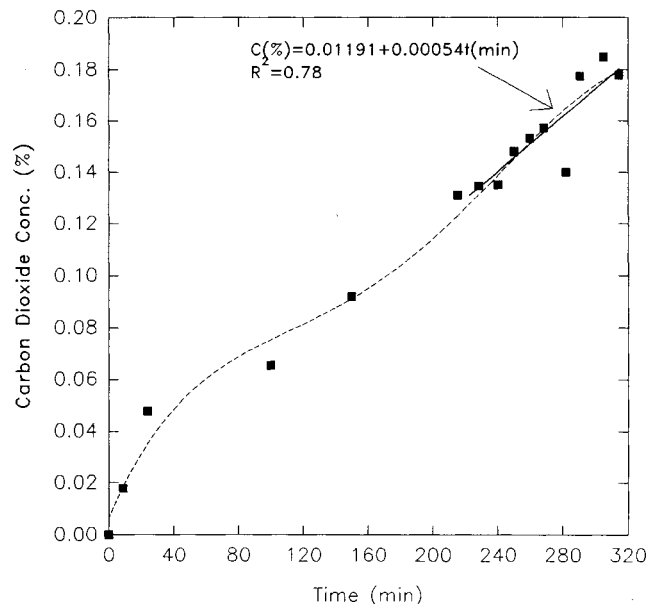


Figure 2—Time profile curve (---) for CO₂ concentration in center chamber of Gilbert and Pegaz cell with regression line (---) noted for steady-state gas transfer.

TABLE 1. Physical and barrier means for corn protein, wheat protein, and cellophane films

Property	Wheat				Com				Cellophane
	n	X	CV	Range	n	X	CV	Range	X
Thickness (µm)	26	140	8.2%	123 - 166	21	89	9.3%	76 - 102	36 *
Tensile strength (MPa)	37	1.8	40.4%	0.4 - 4.5	21	0.4	40.0%	0.1 - 0.9	114 *
Elongation (%)	37	25	32.0%	11 - 41	-	-	-	-	20 *
Tear strength (g/mm)	26	30	15.0%	19 - 32	21	<16	-	-	<16
Bursting strength (kPa)	24	85	7.0%	76 - 99	-	-	-	-	168
H ₂ O permeability ($\frac{\text{pmol}\cdot\text{m}}{\text{m}^2\cdot\text{s}\cdot\text{Pa}}$)	6	69.7	14.5%	57.1 - 88.1	6	22.6	13.0%	20.1 - 28.8	22.6
O ₂ permeability ($\frac{\text{amol}\cdot\text{m}}{\text{m}^2\cdot\text{s}\cdot\text{Pa}}$)	7	3.5	39.0%	2.2 - 6.4	7	34.8	137.4%	0 - 139.2	3.7 - 18.5
CO ₂ permeability ($\frac{\text{amol}\cdot\text{m}}{\text{m}^2\cdot\text{s}\cdot\text{Pa}}$)	4	1.2	244.9%	0 - 4.2	6	216	88.4%	62 - 586	0 *

n = Number of samples.
 \bar{X} = Mean.
 CV = Coefficient of variation.
 1pmol = 1×10^{-12} mol.
 1amol = 1×10^{-18} mol.
 * Values taken from manufacturer's literature.

RESULTS AND DISCUSSION

THICKNESS

A mean thickness value of 140 µm (5.5 mils) was observed for wheat protein films. The wheat protein films produced in this study were thicker than cellophane; it is typically close to 36 µm (1.4 mil) thick when produced uncoated (Flexel, 1989). Corn protein films had a mean thickness of 89 µm (3.5 mils). The depth of the casting mold determined the thickness of these films. Thinner

Wheat films of this study were unable to take much stress once a rip had been formed because of the low tear strength that it possessed but apparently the cellophane was even more susceptible to tear once a rip had been made. Tear strength of corn protein films was so low that it could not be measured using the Elmendorf Tear Tester. Tear strength was less than 16 g/mm since the instrument could not accurately measure below this value. This was another indication that corn zein formed a brittle film in this study.

TABLE 2. Estimates and standard errors for ε (degree/ °C • day) and φ (degree/day), and coefficient of determination R² using equation 3

	1987 (storage T)	1989 (storage T)	1989 (pulp T)
ε (deg / °C • day)	0.1232 (0.0048)	0.0829 (0.0011)	0.0828 (0.0011)
φ (deg / day)	-0.0738 (0.0418)	-0.0209 (0.0149)	-0.0860 (0.0156)
R ²	0.8394	0.9730	0.9731

MODEL APPLICATION TO VARIABLE TEMPERATURES

Using pulp temperature data. First, the applicability of equation 3 to variable temperature tests in 1989 was investigated by applying equation 3 with parameters ε and φ as found for 1989 (pulp temperature case) and by using actual pulp temperature data sets of the 4 variable temperature tests. The standard approach was to assume that hue changes due to temperature were additive regardless of the order of temperature application (i.e., commutativity), and that temperature change had no effect by itself. This assumption meant that the rate of change of Hue H with respect to time under constant storage temperature could also be used for variable temperature conditions. Here, the standard method is based on finite differences and required that a small enough time interval Δt was used during which the storage temperature could be considered to be constant (for our case, Δt was 15 min or

TABLE 3. Mean statistics and t-test for differences between observed and predicted hue angle H using pulp temperature data

	Mean value	Std. error of mean	Max. value	Min. value	T-test sig. level
Hue H dif. (degree)	0.9856	0.0593	23.14	-11.48	0.0001
(107° ≥ observed H ≥ 66°)					

and to reapply equation 3 between t_{i+1} and t_{i+2}, and so on. Hue angle H computed using equation 4 formed the Time Shift data set containing model-based predictions directly paired with actual observations.

Next, a paired-observation t-test was done between the Time Shift data set and the observed Hue angles from all variable temperature treatments, with results shown in Table 3 which showed statistically significant differences over a total of 1,013 observations, although the mean of Hue differences was only 0.99° or 2.5% of the range of observed Hue angles (107 to 66°). Figure 2 is a plot of the difference between predicted and observed hue angle H for each fruit at each test day for all four variable temperature treatments: the vertical bars denote sample standard deviations and the best fit line is also shown. In Table 3, the maximum and minimum differences (23.14 and -11.48) were caused by measurement errors: for example, the 23.14 value corresponded to an observed hue angle H of 74.77°

the clamping device used to hold films in place in the tester.

WATER VAPOR TRANSMISSION

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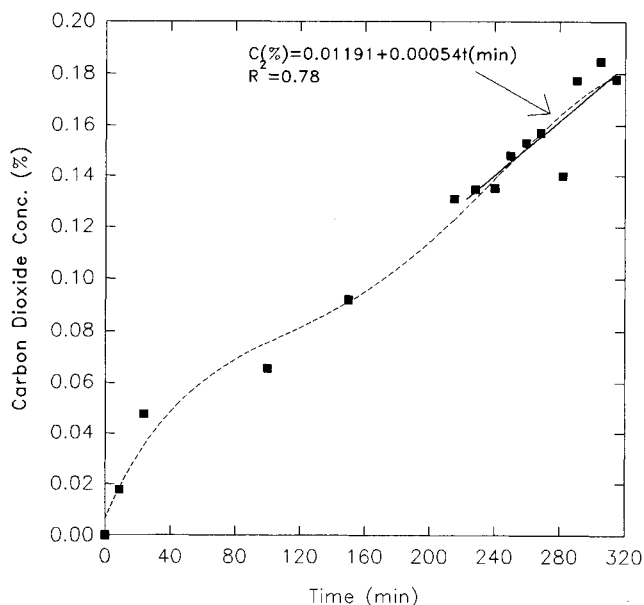


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TENSILE AND ELONGATION

Wheat films were found to have a mean tensile strength of 1.8 MPa (270 lb/in.²). Cellophane, 36-µm thick, has a tensile strength in the machine direction of 114 MPa (16,500 lb/in.²) (Flexel, 1989). This is stronger than any of the wheat films produced. Corn protein films were much weaker than wheat protein films. Mean tensile strength for corn protein films was 0.4 MPa (60 lb/in.²). The low mean tensile strength indicates that corn protein films are fairly brittle.

A mean elongation value of 25%, determined for the wheat films was similar to an elongation in the machine direction of 20% (Flexel, 1989) for cellophane. Brittleness of the corn protein films was even more apparent with elongation measurements. Elongation was so small that it could not be measured. Cellophane and other polymeric films are more elastic than corn zein films. However, Andres (1984) has stated that plasticizers can be added to make corn protein films more flexible by hydrophobically binding zein with plasticizers such as vegetable oils and glycerin.

TEAR

Mean tear strength for wheat protein films was 30 g/mm. Cellophane observed in the laboratory had a lower tear strength than the Elmendorf tester could measure.

Wheat films of this study were unable to take much stress once a rip had been formed because of the low tear strength that it possessed but apparently the cellophane was even more susceptible to tear once a rip had been made. Tear strength of corn protein films was so low that it could not be measured using the Elmendorf Tear Tester. Tear strength was less than 16 g/mm since the instrument could not accurately measure below this value. This was another indication that corn zein formed a brittle film in this study. No attempt was made to strengthen the corn protein films using additives or coatings.

BURSTING STRENGTH

Mean bursting strength of wheat protein films was 85 kPa, ranging from 76 to 99 kPa. Bursting strength of cellophane was observed to be 168 kPa, which is approximately twice the mean bursting strength of the much thicker wheat protein film.

WATER VAPOR TRANSMISSION

Wheat film, mean water vapor permeability of 69.7 pmol·m/m²·s·Pa (2.8×10^4 g·mil / 100 in.²·24 h·atm), was more permeable than cellophane, mean water vapor permeability of 22.6 pmol·m / m²·s·Pa (0.9×10^4 g·mil/100 in.²·24 h·atm). However, it should be noted that these measurements are multiplied by their thickness and the wheat film is nearly four times thicker than the cellophane. Corn zein film had a mean permeability value of 22.6 pmol·m / m²·s·Pa (0.9×10^4 g·mil / 100 in.²·24 h·atm), similar to cellophane water vapor permeability.

OXYGEN GAS TRANSMISSION

The mean O₂ gas permeability for wheat protein films was 3.5 amol·m / m²·s·Pa (2.0 cm³·mil / 100 in.²·24 h·atm). A CV of 39% was believed to be due mainly to the fact that the measured permeabilities were closing in on the lower limits of the testing abilities of the Oxtran.

Cellophane was reported to have an O₂ gas permeability of 3.7 to 18.5 amol·m / m²·s·Pa (2 to 10 cm³·mil / 100 in.²·24 h·atm) at 23.9° C (75° F) and 35% RH (Flexel, 1989). Even though the tests were performed at slightly different temperatures, wheat protein films possess very low O₂ gas permeabilities similar to cellophane.

Corn protein film specimens had a mean oxygen gas permeability of 34.8 amol·m / m²·s·Pa (19.7 cm³·mil / 100 in.²·24 h·atm) at 1 atm., 37.8° C (100° F) and 0% RH. Corn protein films had an O₂ gas permeability range of 0 to 139.2 amol·m / m²·s·Pa (0 to 79 cm³·mil / 100 in.²·24 h·atm). Obviously, these corn protein films had a wide variation yet oxygen gas permeabilities are still relatively low and considered impermeable.

CARBON DIOXIDE GAS TRANSMISSION

All wheat protein films had a CO₂ gas permeability of 0 amol·m / m²·s·Pa except for one which measured 4.2 amol·m / m²·s·Pa (2.24 cm³·mil / 100 in.²·24 h·atm). The Gilbert and Pegaz permeability cell does not have a low level of detectability but it is low enough to give evidence that the wheat protein films performed well as a CO₂ barrier. In comparison, uncoated cellophane is reported to be virtually impermeable to CO₂ gas (Flexel, 1989).

Corn protein films had a mean CO₂ gas permeability of 216 amol·m / m²·s·Pa (117 cm³·mil / 100 in.²·24 h·atm) and ranged from 62 to 586 amol·m / m²·s·Pa (33 to 315 cm³·mil / 100 in.²·24 h·atm). This is a relatively large variation, much the same as corn protein film's O₂ gas permeability. However, corn protein films still show relatively low CO₂ gas permeabilities.

CONCLUSIONS

Conclusions drawn from this study were:

1. The method of production adapted for each type of film yielded sample films that were consistent in measured physical and gas transmission properties.
2. Corn and wheat protein films had low permeabilities for carbon dioxide and oxygen, however, both films had relatively high water vapor permeabilities.
3. Corn and wheat protein films had low tear and tensile strengths, yet the wheat films maintained a larger percent elongation.
4. Cereal-based protein films were thicker than cellophane, however, cellophane maintained a greater tensile value than both.

5. Elongation of the wheat protein films was greater than cellophane while the corn protein films had virtually no elongation.
6. Cellophane exhibited a greater amount of bursting strength than wheat protein films. Bursting strength of corn protein films could not be measured because of their brittleness.

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