

Water Vapor Transmission Rates and Sorption Behavior of Chitosan Films

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ABSTRACT: This study measured the water vapor transmission rates (WVTR) and moisture sorption of chitosan films over a range of water vapor pressures at 25 °C. Films of a constant thickness were made using chitosan with 3 levels of deacetylation. Films were tested at test relative humidity (RH) difference between RH values of 84%, 75%, 69%, 53%, 43%, 33%, 23%, 11%, and 0 at 25 °C using ASTM F1249-90 or ASTM E 96-80. The equilibrium moisture content in the films ranged from 3.7% to 31.8% (dry basis) corresponding to 11% to 84% RH. WVTRs of films increased with increase in water vapor pressure. The mean WVTR ranged from 6.7 to 1146 (g/m²/d) over the range of water vapor pressure from 2.68 (11% RH) to 19.9 mmHg (84% RH). The percentage of deacetylation of chitosan films and the viscosity of the cast solution did not have an effect on the WVTR properties of chitosan films.

Keywords: chitosan, films, WVTR, isotherms

Introduction

IN 1936, GW RIGBY WAS GRANTED A PATENT FOR MAKING FILM from chitosan and a 2nd patent on making fibers from chitosan. The films were described as flexible, tough, transparent, and colorless with a tensile strength of about 9000 psi. Muzzarelli (1974) also described similar procedures for casting films from chitosan. Averbach (1978) concluded that film forming qualities of chitosan were dependent on the structure. His viscosity data did not correlate well with the film forming characteristics (Averbach 1978). The mechanism for and the prediction of water transport through hydrophilic films like chitosan films are extremely complex. The complexity is due to nonlinear water sorption isotherms and water content dependent diffusivities (Schwartzberg 1986). Water vapor transmission of hydrophilic films varies nonlinearly with water vapor partial pressure. If films are cationic and strongly hydrophilic, water interacts with the polymer matrix and increases permeation for water vapor (Pascat 1986).

Muzzarelli (1974) reported a WVTR of 1200 g/m²/d at 100 °F and 90% relative humidity (RH) test difference for chitosan membranes 20 um thick. Wong (1992) measured the water vapor permeability of chitosan and chitosan-lipid films cast from 1% chitosan solution (> 70% deacetylated) using formic acid. These chitosan films had a water vapor permeability (WVP) of 0.41 g mm/m²/d/mmHg tested at unspecified conditions. These films were reported as smooth without pores or cracks. Chitosan films made with plasticizer (glycerine) levels of 0.25 and 0.50 ml/g had a mean WVP of 2.89×10^{-4} g/m²/d/mmHg at 25 °C between 0% to 11% RH (Butler and others 1996). Butler and others (1996) stated that their chitosan films were extremely good barriers to oxygen, while having low water vapor barrier properties because of their hydrophilic nature. Their study concluded that increasing plasticizer concentrations had a negative effect on barrier properties while improving formation, mechanical, and handling properties. They also measured the ethylene permeation of chitosan films. On the average, chitosan films had an ethylene permeability rate of 1.6×10^{-4} cc/m²/d/atm with 0.25 ml/g plasticizer concentration and 3.1×10^{-4} cc/m²/d/atm for a plasticizer concentration of 0.50 ml/g.

Caner et al. (1998) prepared chitosan films using various acid and plasticizer concentrations. They reported measured water vapor permeability coefficients ranging from 1.74×10^{-5} to

7.04×10^{-4} g/m²/d/mmHg at 25 °C between 50% to 100% RH and concluded that storage time had no effect on barrier properties of chitosan films. Presently, there are no other reported WVTR values for chitosan films at various relative humidity conditions or water vapor pressures.

The objective of this study was to produce films from chitosan having 3 levels of deacetylation (91.7%, 84.0%, and 73.0%) and measure their water vapor barrier properties over a range of water vapor pressures. Mathematical models will be developed and tested for statistical differences between the levels of deacetylation for WVTR and moisture sorption data. Also, moisture sorption isotherms of the chitosan film were determined at the specified test temperature and the relationship between the isotherms and their WVTR measurements observed. Models were fitted to the WVTR data to enable calculation of water vapor transport through chitosan films.

Material and Methods

Film Formation

A 2% chitosan solution was prepared using a 1% acetic acid solution. Sixteen g of chitosan (Vanson, Redmond, Wash., U.S.A.) was mixed with 800 ml of distilled water and 8 ml of glacial acetic acid (Mallinckrodt, Paris, Ky., U.S.A.). Three types of chitosan were used in making films (Table 1). The chitosan mixture was homogenized for 10 min at 7,000 RPM using a Virtis homogenizer (Virtis Co., Inc., Gardiner, N.Y., U.S.A.). After homogenizing, the chitosan solution was filtered under a vacuum through 6 to 8 layers of 40-grade cheesecloth. This removed most of the undissolved impurities in the solution. While pulling a vacuum with an aspirator, the vacuum flask containing the chitosan solution was sealed with a rubber stopper and held for a few hours to de-gas the solution to prevent the formation of air bubbles in the films when the casting solvent evaporated.

After degassing, 250 ml of chitosan solution was cast onto a glass plate approximately 34 cm × 34 cm. Film thickness was controlled by pouring a constant amount of chitosan solution onto a level glass plate framed with strips of plastic or wood. The cast film was dried overnight at ambient conditions (23 °C and 50% RH). Ambient temperature and relative humidity varied with the season. A total of 9 glass plates were used to cast films at the same time. After the solvent evaporated, films were removed

and labeled according to the casting plate. Test samples approximately 73 mm \times 73 mm were cut from the cast films. These samples were dried further to obtain a desired dry weight.

Film Thickness

Chitosan film thickness was measured after drying films at 60 °C in a desiccator containing Drierite (W.A. Hommond Drierite Co., Xenia, Ohio, U.S.A.) for 3 d. Films were dried at this relatively low temperature in order not to degrade or change the chitosan structure in the polymer. Initially, dry film weights were measured daily until it was determined that no measurable weight loss was seen between d 3 and 4. Therefore, films were dried for 3 d prior to recording the dry weights of films to the nearest 0.001 g. Thickness was recorded according to ASTM D65 using an electronic micrometer (Model 49 to 60, Testing Machines Inc., Amityville, N.Y., U.S.A.). An average of 3 measurements on each sample (73 mm \times 73 mm) was recorded to the nearest 0.0127 mm.

Moisture Sorption Isotherms

Water sorption isotherms were determined using the method of discontinuous registration of weight changes in a static system (Labuza 1984). Moisture sorption isotherms of chitosan films were determined by placing film samples (73 cm \times 73 cm) into a controlled humidity environment at a constant temperature until equilibrium was obtained.

After drying at 60 °C in a desiccator for 3 d, dry weights of films were measured to the nearest milligram using an electronic scale (Sartorius, Bohemia, N.Y., U.S.A.). The dried film strips were placed into environments with various relative humidities above salt solutions in desiccators (Labuza 1984). The relative humidities were: 11% RH (lithium chloride), 23% RH (potassium acetate), 33% RH (magnesium chloride), 43% RH (potassium carbonate), 53% RH (magnesium nitrate), 69% RH (potassium iodide), 75% RH (sodium chloride), and 84% RH (potassium chloride). Ten film samples cut from each plate were placed into each condition. Desiccators containing the film samples and salt solutions were placed into an environmental chamber (Model 317322, Hotpack, Corp., Philadelphia, Pa., U.S.A.) at 25 °C. Samples were conditioned for 2 wk. The 2 wk sorption equilibrium was determined by weighing samples at 7-d intervals until the sample weight did not change by more than 2 mg of water/g of polymer by dry weight (Labuza 1984). Initially, sample weights were recorded on d 7, 14, and 21. After comparing measurements, it was determined that the weight change was 2 mg of water per gram of polymer or less between d 14 and 21. Therefore, a 2-wk period was used to condition films for testing to assure that the films were in equilibrium sorption. After 2 wk, final weights were measured and final moisture contents calculated. Moisture isotherms were plotted (moisture content dry basis vs water vapor partial pressure). Experimental water sorption isotherms were fitted using a nonlinear (NLIN) regression analysis procedure in SAS (SA 1996). The NLIN procedure uses an iterative modified Gauss-Newton method to regress the residuals onto the partial derivatives of the model until the parameter estimates converge.

Water Vapor Transmission Rates

Two standard test methods were used to determine water vapor transmission rates (WVTR). All film samples were conditioned for 2 wk at the relative humidity or water vapor partial pressure used in the test. The test method used was dictated by the conditions (water vapor partial pressure) of the test. WVTR measurement of 69% RH or more were tested according to ASTM E 96-80 (ASTM 1980). This method measured WVTR using a test cup as described by Butler (1995). The test cups were filled with 20 ml of Drierite (W.A. Hommond Drierite Co.) (desiccant) to produce a 0% RH below the film. A chitosan film (16.3 cm²) was

Table 1—Chitosan materials used to make self-supporting films.

Material	Species	% Deacetylation*	Viscosity of Chitosan Solution(cps)*
VNS-485	Shrimp	91.7	30
VNS-575	Shrimp	84.0	80
K05170904DD	Shrimp	73.0	250

*as measured and reported by the manufacturer, Vanson, Redmond, Washington

mounted to the top of the cup to give an air gap at approximately 0.6 cm between film surface and desiccant. WVTR of chitosan films were measured 53%, 69%, 76%, and 84% RH at 25 °C. After taking initial weights of the test cup, the cups were placed into an environmental chamber with an air velocity rate of 340 ft/min (Model 317322, Hotpack Corp.) at the appropriate relative humidity test conditions (\pm 5% RH) and at 25 °C \pm 2 °C. Weight gain measurements were taken by weighing the test cup to the nearest 0.001 g with an electronic scale (Sartorius Corp.) every h for 8 h. A plot of weight gained versus time was used to determine the WVTR. The slope of the linear portion of this plot represents the steady state amount of water vapor diffusing through the film per unit time (g/h). WVTR was expressed in units of grams per meter squared per d. Steady-state weight gain over time (slope) yielded a regression coefficient of 0.99 or greater. Four samples were used at each test condition.

Chitosan films to be tested for WVTR with a %RH differential between 0% and 53% RH were tested according to ASTM F 1249-90 (ASTM 1990). After conditioning for 2 wk in their conditions, samples were masked using aluminum foil with an adhesive backing (All-Foils, Inc., Brooklyn Heights, Ohio, U.S.A.) with a test area of 5 cm². These samples were tested for WVTR at 25 °C using the Permatran W600 Water Vapor Permeability Tester (Modern Controls, Inc., Minneapolis, Minn., U.S.A.). Five %RH were used, 11%, 23%, 33%, 43%, and 53% RH. Mylar LB 92 used as a reference film gave a WVTR value of 12.5 g/m²/d at 25 °C and 100% RH.

Measured values of WVTR (WVTR_m) were corrected (WVTR_c) for air gap distance between water or dessicant and the film according to procedures outlined in Gennadios and others (1994) and McHugh and others (1993) and tested for statistical significant difference using Analysis of Variance (ANOVA) procedures (SAS 1996).

Statistical Analysis and Experimental Design

The experimental design used was a generalization of the randomized block design called a split-plot design. This multi-factor design is used when a completely randomized order of runs within the block is not possible. Split-plot design is a specialized design for a factorial experiment. The blocks are the 3 replications. Each block is divided into parts called whole plots (main treatments). The 3 main treatments are the types of chitosan polymer used to make the biopolymer films (Table 1). The 3 types of chitosan are referred to as "Product" in my design. Each whole plot is divided into parts called split-plots or subplots which consisted of the 8 relative humidities. The split-plot design recognizes that treatments applied to main plots are subject to larger experimental error than those applied to subplots. Therefore, different variances are used as denominators for the corresponding F ratios (Freund and Littell 1981).

The split plot experiment can be viewed as a particular form of mixed model with 2 random effects (whole plot and subplot errors) and a 2 factor structure of fixed effects (Yandell 1997). The response is partitioned as

$$\text{Response} = (\text{Whole plot}) + (\text{Split plot}),$$

with each part subdivided into mean model and error,

Table 2—Least square means of the percentage of moisture content in chitosan films stored at various relative humidity conditions and 25°C.

Water Vapor Pressure (mmHg)	% Relative Humidity	91.7% DA Chitosan Films (% dry basis)	84.0% DA Chitosan Films (% dry basis)	73.0% DA Chitosan Films (% dry basis)	Combined DA Chitosan Films (% dry basis)
2.68	11	3.42	3.68	4.00	3.70
5.34	23	4.28	5.57	6.31	5.39
7.77	33	6.00	7.97	8.65	7.54
10.2	43	6.55	8.61	9.12	8.09
12.5	53	10.5	13.2	13.7	12.5
16.3	69	15.2	19.4	19.5	18.0
17.8	75	21.4	23.3	25.6	23.4
19.9	84	27.9	32.2	34.1	31.4

DA = Deacetylation

Whole Plot = Mean + Product + Whole plot error

Split plot = RH + RH*Product + Subplot error

The split plot model is

$$Y_{ijkl} = \mu + \alpha_i + Y_{ik} + \beta_j + (\alpha\beta)_{ij} + \varepsilon_{ijk} + S_{ijkl}$$

Where Y_{ijkl} = Response variable, μ = Mean, α_i = Product, Y_{ik} = Whole plot error, β_j = Relative humidity (RH), $(\alpha\beta)_{ij}$ = Product RH Interaction, ε_{ijk} = Subplot error, and S_{ijkl} = Sampling error.

The response variables were WVTR, sorbed moisture (mg of water/ mg of polymer) and film thickness.

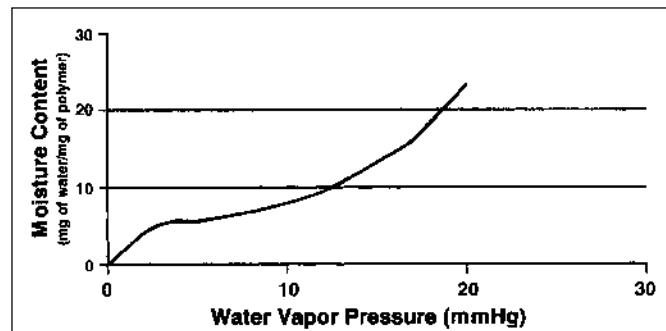
Statistical analysis was performed using the General Linear Models, Least Squared Means, and Non-Linear procedures of SAS (SAS 1996).

Results and Discussion

CHITOSAN FILMS DRIED AT ROOM TEMPERATURE WERE RELATIVELY easy to peel from the glass plates. Increasing air circulation over the films dried them more evenly and quickly. Moisture content of these films dried at ambient were 11 to 15% based on dry basis, and were transparent with a white to slight yellow tint. The yellow tint became more evident as storage relative humidity increased. Dried films were brittle and tended to curl up. Chitosan films became increasingly more flexible and less brittle when stored at high relative humidity.

Moisture Sorption Isotherms

Moisture sorption isotherms of the chitosan films were sigmoidal (Figure 1). A significant ($\alpha = 0.05$) film to water vapor pres-

**Figure 1—The shape of the moisture sorption isotherm for chitosan films at 25°C****Table 3—Least-square means of the water vapor transmission rates of chitosan films at 25°C.**

% Relative Humidity	Vapor Pressure Difference (mmHg)	91.7% DA Chitosan Films (g/m²/d)	84.0% DA Chitosan Films (g/m²/d)	73.0% DA Chitosan Films (g/m²/d)
11	2.68	5.21	6.66	7.70
23	5.34	14.4	14.2	14.6
33	7.77	39.6	39.2	32.5
43	10.2	139	126	117
53	12.5	305	288	259
69	16.3	364	437	711
75	17.8	574	668	912
84	19.9	999	1068	1355

sure interaction was obtained (General Linear Model [GLM] method of analysis of variance, SAS 1996). The least squares means (LSMEANS) are reported in Table 2. The LSMEANS of the percentage of equilibrium moisture against deacetylation was not statistically significant. Equilibrium moisture of chitosan films ranged from 3.7% to 31.8% at 11% to 84% RH (2.68 mmHg to 19.9 mmHg). The nonlinear models for each film type was fitted with an exponential equation over the test range of 11–84% relative humidity (Figure 2).

The sorption isotherm is expressed as

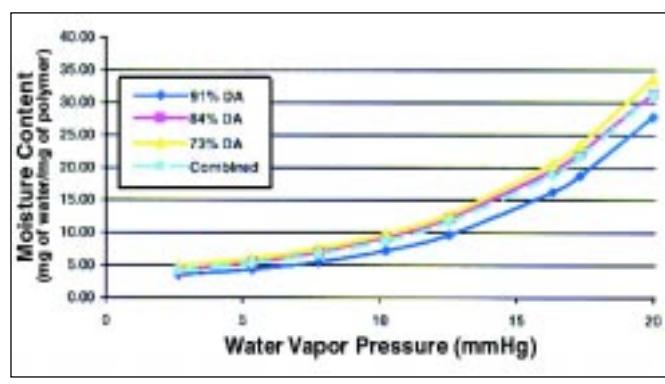
$$y = a + b \exp^{cx}$$

y being the equilibrium moisture (mg of water/ mg of polymer) and x the water vapor pressure (mmHg). Values of a, b, and c are respectfully 2.00, 1.48, and 0.15.

The nonlinear model is graphically plotted in Figure 2. Analysis of variance between film types (deacetylation) showed no significant difference between a combined fitted exponential curve and that of separate fitted curves. Therefore, the data can be described by 1 expression.

Water Vapor Transmission Rates

Water vapor transmission rates increased with an increase in water vapor pressure. The LSMEAN WVTR ranged from 5.2 to 1354 g/m²/d as water vapor pressure increased from 2.68 to 19.9 mmHg (Table 3). There was no significant difference in WVTR between the types of chitosan film. The percentage of deacetylation of chitosan films and the viscosity of the cast solution did not have an effect on the WVTR properties of chitosan films. Therefore, a single nonlinear model was applied to all points (Figure 3). There were 2 exponential equations derived to ex-

**Figure 2—Moisture sorption isotherm models for chitosan films at 25°C**

press this model. A nonlinear model was fitted between 11% and 53% relative humidity, which represents WVTR measurements made using the method ASTM F1249-90. This equation is

$$y = -6.32 + 3.17 \exp^{0.36(x)}$$

with y being WVTR ($\text{g/m}^2/\text{d}$) and x is the water vapor pressure difference (mmHg). Another exponential fit was performed between 53% and 84% relative humidity using measurements from ASTM E96-80 test procedures. This expression is

$$y = 147 + 4.55 \exp^{0.27(x)}$$

The average thickness of chitosan films used for sorption isotherms was $40.1 \mu\text{m}$ with a standard deviation (std. dev.) of $4.6 \mu\text{m}$. WVTR test films thickness had a mean of $39.6 \mu\text{m} \pm 3.6 \mu\text{m}$ (std. dev.). The exponential model equations can be used in the thickness ranges stated to determine WVTR when exposed to conditions of this study or moisture content of chitosan film if water vapor pressure (mmHg) is known. But the main purpose of these equations was to allow for statistical testing for significance and to graphically display water vapor dependency on measured response variables.

WVTR_c values are given in Table 4. ANOVA between the nonlinear models of WVTR_m and WVTR_c resulted in no significant difference between the measured and corrected WVTR values.

Moisture sorption isotherms of cellulosic films and also for most foodstuffs are basically sigmoidal in shape. This type of sorption isotherm also characterizes chitosan films. Moisture sorption increases rapidly at the low water vapor pressures while increasing moderately at intermediate pressures from 7.0 to 12.5 mmHg. At high water vapor pressures, the moisture sorption increase becomes exponential. Moisture content of chitosan films at the higher water vapor pressures between 16.3 mmHg and 19.9 mmHg was as high as 20 to 30 % on a dry basis. With such a large gain in moisture, one would suspect swelling to occur at some point. Swelling would cause conformational change in the microstructure of the film that would not only increase moisture sorption, but also open up the polymer structure to allow an increase in permeant flux. Swelling results in deviations from "Fickian" behavior (Rogers 1985). A consequence of swelling is changes in the polymer structure that occur in response to stresses generated within the film during sorption (Rogers 1985). This increase of water vapor solubility leads to an increase in water vapor permeability. Water vapor acts as a plasticizer inside the chitosan film's matrix. This is evident through the observable increase in flexibility and decrease in water vapor barrier properties through the plasticizing effect of water causing the polymer

Table 4—Least-square means of the measured WVTR and the corrected values of WVTR.

% Relative Humidity	Vapor Pressure Difference (mmHg)*	*WVTR _m Chitosan Films (g/m ² /d)	**WVTR _c Chitosan Films (g/m ² /d)	%Difference in WVTR
11	2.68	6.65	6.69	0.60
23	5.34	14.2	14.5	2.11
33	7.77	36.5	37.0	1.37
43	10.2	126	130	3.17
53	12.5	278	296	-5.76
69	16.3	504	464	-7.94
75	17.8	719	647	-10.0
84	19.9	1140	984	-13.7

*measured water vapor transmission rates

**corrected water vapor transmission rates

structure to become looser. Upon further research, swelling will be quantified. This will allow for better calculation of permeability and diffusion coefficients of chitosan films to water vapor at 25 °C.

When comparing sorption isotherms (Figure 2) to the graph of WVTR as compared with water vapor pressure, at lower pressures, water sorption is the predominate process with water vapor transmission rate through the film becoming increasingly significant at water vapor pressures above 10 mmHg. At higher water vapor pressures, the rate of increase is greater in WVTR than in the rate of moisture sorption. This suggests that more than polymer-penetrant interactions are occurring. Penetrant-penetrant interaction and the formation of penetrant multilayers that leads to mass flow of permeant result in large increases of WVTR. The shape of the moisture sorption isotherm correlates well to that of the WVTR dependency on water vapor pressure. The difference being that at low water vapor pressures the moisture sorption isotherm is concave to the X-axis (pressure) while WVTR is convex to the pressure axis.

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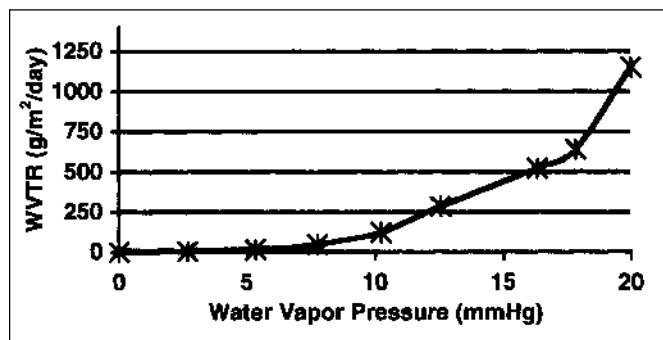


Figure 3—The dependency of water vapor transmission rate on water vapor pressure for chitosan films at 25 °C

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MS 19991059

Technical Contribution No. 4566 of the South Carolina Agric. and Forestry Research System

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