Physical and mechanical properties of alginate edible films formulated with a two level experimental design

M. Rangel-Marrón, C. Montalvo-Paquini, E. Palou, and A. López-Malo

***Abstract*—** The present study focuses on the effects of sodium alginate (0.5-2.5% w/v) and glycerol (0.5-1.5% w/v) concentration and the film forming method (FFM), dry (D) or wet (W) on physical (moisture content, thickness, water solubility (WS), water vapor permeability (WVP), and color) and mechanical properties of edible films. The effects were formulated and analyzed using a two-level factorial design. Prediction equations were obtained and optimized for each response variable. To generate insoluble polymers 2% of $CaCl_2$ was added directly to the film emulsion (wet method). For the dry method, the emulsions were dried at 60 ºC for 6 hours, and then $CaCl_2$ was added. The moisture content, water solubility, thickness, hue angle, chroma, luminosity, and percentage elongation were significantly affected ($p < 0.05$) by the evaluated factor. Furthermore, glycerol concentration did not affect the water vapor permeability and alginate concentration did not affect the puncture strength. The films obtained by wet method, showed the highest water solubility values 66.06% (0.5% alginate-glycerol) and WVP 579.74 g mm/kPa h m^2 (2.5 - 0.5% glycerol alginate). Unlike, the films formed by the dry method, the water solubility was 44.66% and WVP was 13.66 g mm/kPa h m^2 for the 0.5 - 1.5 and 2.5 - 0.5% alginate-glycerol concentrations respectively. Predictive equations for each response variable showed a good fit of the experimental data ($R^2 > 0.999$). Optimization results suggested that alginate 2% and glycerol 1.5% concentrations and dry method can be considered a better formulation for edible films.

***Keywords*—** Edible films, glycerol, mechanical properties, physical properties, sodium alginate.

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I. INTRODUCTION

CONSUMERS demand higher quality and longer shelf-life in foods while reducing disposable packaging materials and increasing recyclability [1]. One of the primary and important ways is to use biodegradable packaging materials. Many researchers as well as worldwide companies are now emphasizing on the development of ecofriendly packaging solutions by using the ecological advantages of biopolymers in food packaging applications [2]. These materials have many properties such as: biodegradability, availability or obtained from renewable resources and very good potential to replace synthetic polymers, make them ideal for wide use in both common and especial applications; including potential to replace synthetic polymers [3], [4]. Biodegradable packaging is made entirely from natural materials such as carbohydrates (alginate, pectin, starches, cellulose, lactose, sucrose) and/or proteins (whey, casein, collagen or hydrolyzed protein); this materials can be recycled and no additional energy is required to break them down [5], [6]. Renewable biopolymers have been increased interests in edible films research. Films and coatings have been used to reduce spoilage during processing and storage of fresh-cut fruits. Edible films provide a semi permeable barrier that helps to extend shelf life by reducing the migration of moisture, the loss of solutes from the fruit, as well as the respiration and oxidation reactions [7]-[9].

The alginate is a salt of alginic acid, which is a mixture of polyuronic acids, isolated from the cell walls of a number of species from brown seaweeds (Phaeophyceae). This polysaccharide is formed by acid monomers β-D-mannuronic acid and α-L-guluronic, being a non-toxic polysaccharide [10], [11]. This biopolymer has interesting functional properties as its colloidal properties and thickeners, helps to stabilize suspensions and emulsions; and has the ability to form films and gels [12]. Alginate is capable of forming an insoluble gel or polymer resistant in the presence from polyvalent metal cations such as calcium. Calcium is preferred to crosslink alginate for biomedical applications due to the mild reactions conditions [13]. The gelling mechanism is based on interactions between the calcium ions and carboxylic groups, forming a three-dimensional crosslinked network. This

interaction occurs when mixing components (alginate-calcium) to form a film or by pouring a solution from calcium on alginate layer previously dried [14].

A.E. Pavlath, C. Gossett, W. Camirand, G.H. Robertson [15] reported based edible films alginate slowly dissolves when they are immersed in a solution of multivalent ions (calcium and zinc). Moreover, edible films containing hydrocolloids such as alginate do not perform as well as a moisture barrier for hydrophilic in nature, since the flow from water vapor through the film is not linear with gradient the partial pressure of water vapor [1].

Plasticizers such as glycerol are necessary to form the basis from edible polysaccharide films, since they can increase the flexibility of the film due to the decrease of hydrogen bonds between polymer chains, resulting increased of intermolecular space. Therefore, it can increase the permeability of the film to oxygen and moisture [16]. Several studies [7,8,14,17,18] had been focused primarily on film formation of alginate based edible by pouring the mixture (alginate-glycerol) obtaining the polymer by adding calcium chloride, and subsequently the emulsion subjected to a relative humidity generated pre-set (generally low), to remove the solvent (water) and obtaining the edible film. However, there is little information on the effect of dewatering the mixture (alginate-glycerol) and then adding calcium chloride to the film formation.

The factorial design is a method that is often used to speed up the task´s solution. It has been applied in various branches of science and industry [19]. Furthermore, the optimization has been used in food engineering and an efficient operating system and unitary process in order to obtain a highly acceptable solution [20].

The objective of this study was 1) to determine the effect of sodium alginate (0.5-2.5% w/v) and glycerol (0.5-1.5% w/v) concentrations, and the film forming method (dry or wet) on physical and mechanical properties of edible films, and 2) to obtain prediction equations and optimize the response variables.

II. Materials and Methods

A. Materials

Alginate (Biopolymer Mexico), glycerol used as a plasticizer, was from Merck (Inc. Corp. Whitehouse Station, NJ, USA) and calcium chloride (PRM, Mexico).

B. Film preparation

Alginate (Biopolymer Mexico), glycerol used as a plasticizer, was from Merck (Inc. Corp. Whitehouse Station, NJ, USA) and calcium chloride (PRM, Mexico).

C. Formation and conditioning of films

The edible films were prepared by adding 12 grams of film forming solution in Petri boxes of 10 cm in diameter previously identified. The first batch was incubated in an oven at 60 ºC for 6 h, at the end, calcium chloride were added (15mL) 2% (w/v) (dry film formulation), while in the second is immediately added calcium chloride (wet formulation). Then, they were stored at a relative humidity of 62% at 25 ºC for 3 hours, and conditioned at 33% relative humidity for analyzing physical and mechanical properties.

D. Physical properties

Moisture content

Moisture content of the films was determined by the loss of weight of the film after drying at 105 ºC for 24 h [21].

Water solubility (WS)

Film solubility was determined as follows: three randomly selected samples (boxes 2 x 2 cm^2) from each type of film were first dried at 105ºC for 24 h. Then, samples were immersed in 50 mL of distilled water and kept in a bath with constant shaking (Yamato Scientific model BT 25, Tokyo, Japan) at 25 ºC for 24 h, after the samples were filtered and dried at 105 ºC for 24 h to determine dry matter not dissolved in water [21]. The rate of solubility was obtained by the following expression:

$$\%WS = \left(\frac{W_{msi} - W_{msf}}{W_{msi}} \right) \times 100 \quad (1)$$

Where: W_{msi} is the initial weight of the dry matter and W_{msf} the dry matter weight of the dispersion process after 24 h.

Thickness

A micrometer (Starrett No. 208, USA) was used to determine the thickness of films formed. Measuring was realized at four different points from film (mm).

Water vapor permeability (WVP)

The permeability of the films was determined gravimetrically using a modified version of standard method ASTM -E96-93 for alginate films [22], [23]. Edible films were cut into 5 cm diameter, then, were placed on glass jar being adjusted on the circumference with parafilm.

The glass jar containing 5 mL of distilled water, leaving an inch air space between the water surface and the film. The above system was stored in dessicator containing a saturated solution of $MgCl_2.6H_20$ at 33% relative humidity at 25ºC. Measurements were recorded over an 8 hours period with

intervals of 60 min. First transfer was obtained of water vapor transmission rate (WVTR) through the slope of the regression analysis of weight loss as a function of time (g/s) and related to the water vapor permeability (WVP) [24] according to the following expressions:

$$WVTR = m_1 / A = g / m^2 s \quad (2)$$

$$WVP = LxWVTR /(p_1 - p_a) \quad (3)$$

Where m_1 is the slope of the weight loss versus time (g/s), A is the exposed area; p_i and p_a are respectively the partial pressures of water vapor in the air and air saturated to 33% relative humidity at 25 ºC. L is the average thickness (mm).

Color

Color parameters (L^*, a^*, and b^*) of alginate edible films were determined by using a colorimeter (Colorgard System/05) calibrated with a white standard plate (L^*= 92.89, a^*= -1.06 and b^*= 0.82). Readings were obtained in the CIELAB scale; Hue angle (h^*) value was calculated by $\tan^{-1}$ (b^*/a^*), and chroma (C^*) by $(a^{*2} + b^{*2})^{1/2}$. Color was determined by triplicate at different locations for each film placed on a circular support (1 cm diameter).

E. Mechanical properties

The mechanical properties of the films were evaluated by puncture test and percentage of elongation [25] A texture analyzer (Texture Technologies Corp., NY, EE.UU.) with a spherical puncturing probe (diameter 1.4 cm) was employed. The film was placed in a holder with a circular hole (r = 0.8 cm). The probe was driven through the film with a speed of 1 mm/s and force displacement curves were recorded through a 25 N load cell. The puncture strength and percentage of elongation was calculated as in (4) and (5). The average of three measurements was performed.

$$Puncture \quad strength = \frac{F\,max}{A_{cs}} \quad (4)$$

Where F_{max} is the maximum applied force (N), A_{cs} is the cross-sectional area of the edge of the film located in the path of the circular gap. $A_{cs} = 2r\delta$, where r is the radios of the hole and δ is the thickness of the film (mm).

$$\%Elongation = \left(\frac{\sqrt{r^2 + d^2} - r}{r} \right) \times 100 \quad (5)$$

Where r is the radius of the film exposed in the circular gap of the film holder (mm) and d represents the displacement of the probe from the point of contact to the point of puncture (mm).

F. Experimental design

To evaluate the effect of alginate concentrations (0.5 or 2.5% w/v), glycerol (0.5 or 1.5% w/v) and the method of forming the edible film (dry or wet) on the response variables (thickness, moisture content, water solubility, water vapor permeability, color, puncture strength, and percentage of elongation) a two level factorial design (2^3), with three replicates generated by Minitab 16.0 statistical software (Minitab, Inc., State College, PA, USA) in a random order, was used (Table. I). The significance of the factors evaluated on each physical property is selected according to $p < 0.05$.

Table. I Formulations of edible films

Treatment	Alginate (%)	Glycerol (%)	FFM[1]
1	0.5	0.5	W
2	0.5	0.5	D
3	2.5	0.5	W
4	2.5	0.5	D
5	0.5	1.5	W
6	0.5	1.5	D
7	2.5	1.5	W
8	2.5	1.5	D

[1] FFM: film forming method, W: weat, D: dry

G. Response model and optimization

The experiments described in Table. I, were conducted at random to obtain the coefficients and the interactions of the evaluated effects. The following expression, contain the factors used to predict and optimize the response variables studied.

$$y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3 \quad (6)$$

Where y is the predicted response, β_0 is the estimated regression coefficient, β_1, β_2, β_3 are the coefficients of alginate, glycerol, and FFM, β_{12}, β_{13}, β_{23} and β_{123} are double and triple interactions of the main effects.

For the prediction of responses studied was used Microsoft Office Excel 2010. The predictive equations were evaluated in terms of the statistical error by the percentage of the root mean square (% RMS) differences between the predicted values and the predicted experimental thickness, moisture content, water solubility, water vapor permeability, puncture strength, and percentage of elongation, root mean square must be less than 10% in order to be considered acceptable and the model is expressed as:

$$\%RMS = \left(\sqrt{\frac{1}{n} \sum_{i=1} \left(\frac{M_{exp} - M_{pre}}{M_{exp}} \right)^2} \right) x100 \quad (7)$$

Where n is equal to the number of observations, the experimental value is M_{exp} responses studied M_{pre} represents the predicted responses [26].

III. Results

A. Moisture content

It was found that the moisture content decreases in the wet method, when the alginate and glycerol concentration increases. Furthermore, in the dry method when alginate and glycerol decreases, the moisture content increases. Because, the mechanism for forming the polymer-polysaccharide interactions glycerol and glycerol-water, change the physical properties of the film [27] as demonstrated by statistical analysis (Table. IV), where the main effects, double interactions and triple interactions significantly affect the moisture content of the film ($p < 0.05$).

Fig. 1 shows that the edible films obtained with the wet method (treatment 1), 0.5% alginate and 0.5% glycerol showed the highest moisture content (97.20 ± 0.33%) compared with the other formulations; this is due to low concentrations of polysaccharide which allow greater availability of free water to participate in the polymerization reactions. Unlike the dry method where an inverse behavior was observed at the same concentrations of alginate and glycerol, obtaining the lowest moisture content (33.48 ± 2.62%). This behavior can be attributed the fact when preparing edible films with the dry method at concentrations of 2.5% alginate and 1.5% glycerol (treatment 8), the calcium chloride promotes the interaction of the carboxyl groups of the alginate to form a polymeric structure more easily retain the water.

When increasing glycerol concentrations, it increases the moisture content because of its water holding capacity. Similar results based edible films were obtained from hydrocolloids psyllium (Plantago seeds of the plant) [28] and potato starch [29], where authors observed that when glycerol concentrations increase significantly increases the moisture content of the films elaborate.

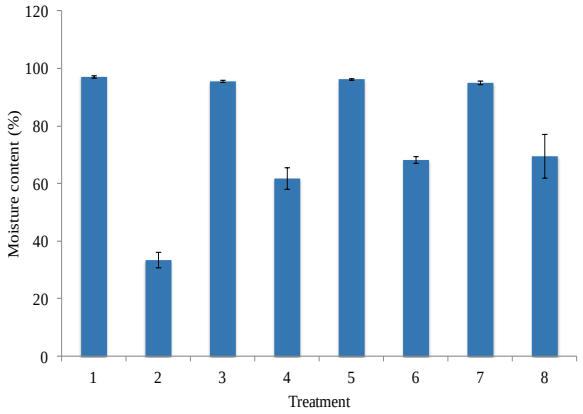

Fig. 1 Effects of alginate-glycerol concentrations and film forming method (Table 1) on edible films moisture content.

B. Water solubility

The method for the wet treatment had a higher solubility of edible films, regardless of the alginate and glycerol concentrations (Fig. 2). With respect to the dry method, the solubility of edible films increases as the concentration of glycerol is added, by increasing the concentration of the plasticizer it decreases intermolecular forces by interacting with the functional groups of the polysaccharide, causing an increase in solubility. It was also noted that higher concentrations of alginate (2.5%) and glycerol (1.5%) (Treatment 8) favor the stability of the film formed.

E. Mwesigwa, A. W. Basit, G. Buckton [30] attributed to the increased solubility of edible films based on polysaccharides, due to the water affinity of the polar groups, so cohesiveness generated is greater than the attractive forces present water-water. The main effects, double interaction (glycerol-forming method of the film) and the triple interaction are shown to be significant ($p < 0.05$) in the solubility of edible film (Table. IV).

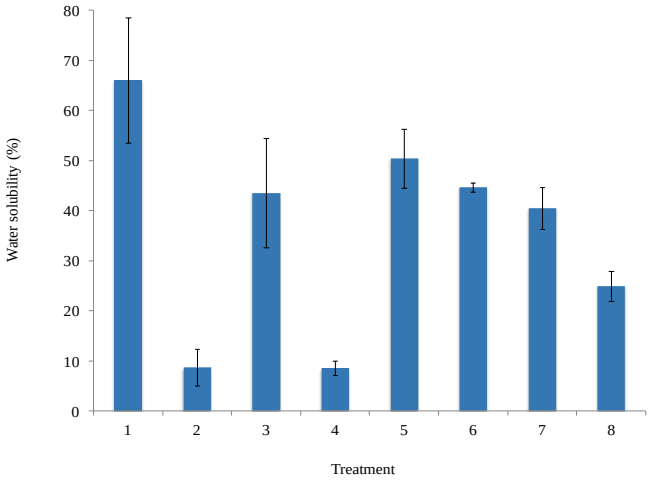

Fig. 2 Effects of alginate-glycerol concentrations, and film forming method (Table 1) on edible film water solubility.

C. Thickness

Table. II shows the mean of triplicates of thickness obtained for each of the 8 treatments evaluated according to the experimental design (factor 2^3). It was observed that less thickness was obtained (0.02 mm) of edible films, when using the dry method and low concentrations (0.5%) and glycerol alginate, unlike the wet method to the same concentrations of polysaccharide and plasticizer, where was obtained a thickness of 0.69 ± 0.13mm (Table. II). While the maximum thickness achieved corresponded to the wet method (2.5% alginate and 1.5% glycerol) obtaining 3.7 ± 1.15 mm. Similar results were obtained for Ionomeric films of alginic acid [15] who assume that when submerged based films alginate calcium chloride occurring two competing reactions occur during the formation of the polymer: 1) dissolution of the alginate in solution and 2) the insolubilization of the film by crosslinking entity Ca^{2+} and carboxylic groups, generating an increase in the film thickness.

The main effects and interactions double (alginate-FFM) and (glycerol-FFM) significantly affect the edible film thickness regardless to the concentrations used (Table. IV).

Table. II Experimental values of the response variables evaluated in edible films.

Treatment	Thickness (mm)	WVP[1] g mm/kPa h m^2
1	0.69 ± 0.13	57.95 ± 16.41
2	0.02 ± 0.00	0.08 ± 0.01
3	2.04 ± 0.46	579.74 ± 182.89
4	0.24 ± 0.12	13.66 ± 2.50
5	1.36 ± 0.03	30.99 ± 2.94
6	0.04 ± 0.03	0.52 ± 0.60
7	3.07 ± 1.15	313.75 ± 163.09
8	0.18 ± 0.02	6.07 ± 0.69

[1]WVP: water vapor permeability

D. Water vapor permeability

Table. II shows that a higher concentration of alginate (2.5%) and glycerol (0.5%) with edible films obtained by the dry method had higher water vapor permeability (13.66 mm ± 2.50 g / h m^2 kPa). In this case the alginate being of hydrophilic nature helps to increase the permeability of the edible film being ineffective as moisture barrier. Statistical analysis showed that the main effects (alginate and FFM) and double interaction (alginate-FFM) are shown to be significant ($p < 0.05$) in the water vapor permeability (Table. IV). Edible films, produced with the wet method had the highest water vapor permeability (30.99 - 579.74 g mm / kPa h m^2) since during the generation of three-dimensional polymer network formed by calcium chloride trapping water molecules, which act as a plasticizer in the crystal lattice, reducing the number of intermolecular bonds in the polymer chain by facilitating the transfer of water vapor through the film [31]. A similar effect was reported for alginate film with oregano essential oil [32].

E. Color

The results of the color measurements performed on the alginate edible films were expressed in accordance with the CIELAB system. Table. III presents the results of the hue angle, chroma, and luminosity (L^*) values. The films obtained for the dry method were slightly opaque. In comparison with the films obtained with wet method, they were translucent, and flexible. In general, hue angle obtained was around bluish green (180º). The results obtained for hue angle were increased when 0.5% alginate-0.5% glycerol (Treatment 1 and 2) were used. Furthermore, when alginate and glycerol concentrations increased (Treatment 8) no significant difference was found ($p>0.05$). A high chroma color may be described as highly saturated, such as the bright red, yellow, blue, etc. Chromaticity is a combination of a color´s hue and chroma [33].

Table. III shows chroma values of edible films. Chroma values ranged from 0.50 in treatment 1 to 1.96 in treatment 8. We observed that, the bluish green chroma (tonality) is brighter when alginate concentration increased from 0.5 to 2.5% and 0.5 to 1.5% for glycerol concentration and FFM for edible films (Treatments 7 and 8). Consequently, chroma values were significantly affected ($p<0.05$) by the main effects. Also, the bluish green for treatments 1 and 2 is less saturated with the decreasing alginate and glycerol concentrations at 0.5%. Table. III shows the L^* values for edible films based on alginate and glycerol concentrations in accordance with FFM (wet and dry). In general, we observed that, brightness was not sufficient changes between treatments. At lower alginate and glycerol concentrations (Treatments 1 and 2), L^* values are brighter than 7 and 8 treatments (alginate (2.5%) – glycerol (1.5%)), respectively regardless of the FFM. Similar results have been reported for alginate film with different degrees of crosslinking and alginate films incorporated with oregano essential oil [32], the authors reported that, the incorporation of $CaCO_3$ did not significantly ($p<0.05$) affect the color of the alginate films. Also they observed that, the low concentration of calcium ions used in the internal gelation of the alginate films was not sufficient to change the light scattering in the polymeric matrix.

Table. III Effect of alginate-glycerol concentrations and film forming method (Table 1) on Hue angle, chroma, and L^* values of alginate films.

Treatment	Hue angle	Chroma	L^*
1	179.11 ± 0.12	0.50 ± 0.29	21.96 ± 0.00
2	179.21 ± 0.03	0.63 ± 0.30	21.84 ± 0.02
3	178.56 ± 0.00	0.76 ± 0.00	21.75 ± 0.02
4	178.52 ± 0.03	1.58 ± 0.23	21.70 ± 0.01
5	178.53 ± 0.07	1.61 ± 0.23	21.70 ± 0.07
6	178.51 ± 0.06	1.83 ± 0.17	21.74 ± 0.07
7	178.55 ± 0.08	2.13 ± 0.34	21.60 ± 0.02
8	178.55 ± 0.06	1.96 ± 0.30	21.62 ± 0.02

F. Mechanical properties

Mechanical properties of edible films are directly related to their chemical structure [34], and may be subjected to various types of stress during their use. Fig. 5 presents the results of the puncture strength of the evaluated edible films. We observed that, the films performed with the wet method, showed lower puncture strength values (< 0.611 MPa) than the edible films prepared by the dry method. This behavior may be attributed to that during the film formation of three-dimensional polymer network formed by calcium chloride, trapping water molecules, which contribute to decrease the puncture strength [35], [36]. We found that, the edible films obtained by the wet method with the concentration of treatment 3 (alginate (2.5%) – glycerol (0.5%)) presented a puncture strength of 0.611 MPa; it was the major value despite having the lowest alginate concentration.

Furthermore, with the films processed by the dry method, we observed that, treatments 2 (alginate, 0.5%-glycerol, 0.5%) and 4 (alginate, 2.5%-glycerol, 0.5%) showed the higher puncture strength, however, there is not significant difference between them ($p<0.05$). Therefore, the glycerol concentration and FFM have significant effect ($p<0.05$) on the puncture strength (Table. IV). The results for treatment 8 suggest that, as the glycerol concentration increase in the edible films the puncture strength decreases and the percentage elongation increases. Glycerol is low molecular weight hydrophilic molecule that could easily establish hydrogen bonding. As a consequence, the density of intermolecular interaction in material decreased and the free volume between polymer chains increased [37]. Furthermore, the puncture strength was affected statistically by the interactions of alginate-glycerol and alginate-FFM, and the triple interaction ($p<0.05$) as in Table. IV.

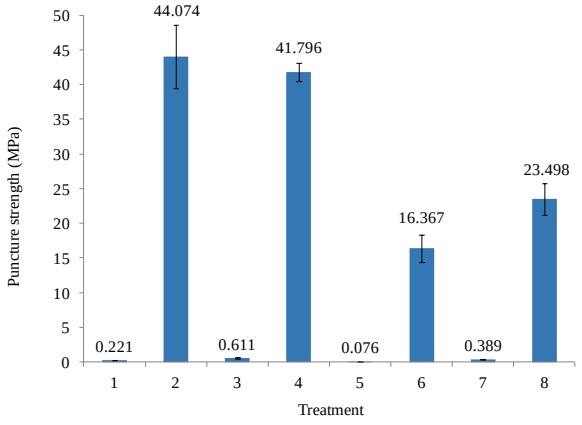

Fig. 5 Effects of alginate-glycerol concentrations and film forming method (Table 1) on edible film puncture strength.

The percentage elongation is the maximum stress supported by the film before breaking and indicates the film´s flexibility. These mechanical properties provided integrity of the edible coatings, because are directly related to their chemical structure [32], [36], [37]. The results obtained for the edible films by the wet method (Treatments 1, 3, 5, and 7) are not different. Also, the films obtained greater elongation but lower puncture strength

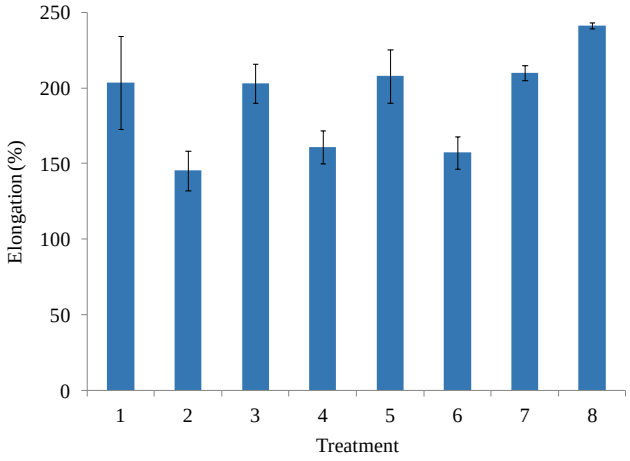

Fig. 6 Effects of alginate-glycerol concentrations and film forming method (Table 1) on edible film elongation

The films obtained by the dry method (treatments 2, 4, and 6) showed that as the puncture strength increases, the percentage elongation decreases, a contrary effect was observed for the treatment 8, in this case, the alginate concentration (2.5%) and glycerol concentration (1.5%), generated a lower puncture strength and higher elongation (241.1%) due to significant effect ($p<0.05$) glycerol concentration.

Similar results were obtained for alginate films [14], and alginate films incorporated with oregano essential oil [32], where the puncture strength increases, while percentage elongation decreases. This phenomenon is explained by the development of cross-linking between carboxyl group of alginate and Ca^{2+}. Statistical analysis support that, the main effects, double and the triple interactions have a significant effect on the elongation percentage of edible films ($p<0.05$) (Table. IV).

G. Prediction and optimization

Table. V shows the ratios obtained with the experimental design (2^3) of the response variables predicted: thickness, moisture content, water solubility, WVP, puncture strength, and percentage elongation.

Prediction equations obtained for each response showed correlation coefficients (R^2) greater than 0.999. The percentage of the root mean square (% RMS) between predicted and experimental values for the thickness, moisture content, water solubility, water vapor permeability, puncture strength, and percentage elongation is less than 1%, indicating that the proposed models are suitable to describe and predict the effects of the factors on the evaluated variables.

Table. IV Estimated probabilities for the effects of the evaluated factors of the response variables.

Term	Moisture content	Water solubility	Thickness	WVP[1]	Puncture strength	Elongation
Constant	0.000	0.000	0.000	0.000	0.000	0.000
Alginate (%)	0.000	0.000	0.000	0.000	0.07*	0.001
Glycerol (%)	0.000	0.007	0.036	0.065*	0.000	0.001
FFM[2]	0.000	0.000	0.000	0.000	0.000	0.000
Alginate (%) * Glycerol (%)	0.000	0.528*	0.706*	0.123*	0.005	0.015
Alginate (%) * FFM	0.000	0.263*	0.002	0.000	0.165*	0.002
Glycerol (%) * FFM	0.000	0.000	0.029	0.078*	0.000	0.007
Alginate (%) * Glycerol (%) * FFM	0.000	0.009	0.55*	0.147*	0.005	0.022

[1]WVP: water vapor permeability, [2] FFM: film forming method

* Not significant at p>0.05

Table. V Coefficients estimated uncoded response variables predicted.

Term	Moisture content	Water solubility	Thickness	WVP[1]	Puncture strength	Elongation
Constant	51.873	34.689	0.002	-46.717	29.929	172.764
Alginate (%)	10.007	-4.786	0.358	164.724	-1.638 *	-4.992
Glycerol (%)	20.254	11.031	0.310	17.622 *	-15.092	-0.690
FFM[2]	-46.245	-46.405	-0.062	42.192	29.743	-28.804
Alginate (%)*Glycerol (%)	-6.636	-1.747 *	0.070 *	-61.764 *	2.333	17.654
Alginate (%)*FFM	10.926	9.629 *	-0.226	-155.936	-1.853 *	-4.213
Glycerol (%)*FFM	21.274	29.840	-0.269	-15.185 *	-14.967	-4.518
Alginate (%)*Glycerol (%)*FFM	-6.860	-8.060	-0.110 *	57.756 *	2.372	16.476

[1]WVP: water vapor permeability, 2 FFM: film forming method

* Not significant at p>0.05

It was determined that the concentrations of sodium alginate (0.5-2.5% w / v), glycerol (0.5-1.5% w / v), and the FFM (dry or wet) had a significant effect ($p < 0.05$) on the content moisture, water solubility, thickness, and percentage elongation of edible films made. The double interaction (alginate-glycerol) was only significant ($p < 0.05$) for moisture content and puncture strength. Alginate-FFM interaction did not affect the solubility and puncture strength of the film, but neither on the other physical properties evaluated. The glycerol-FFM ratio did not influence the water vapor permeability, while alginate-glycerol-FFM had an effect in the moisture content, water solubility, puncture strength, and percentage elongation (Table. V).

For optimization of the evaluated responses, the criteria of minimizing the thickness, moisture content, water solubility, and WVP were established, in addition the percentage elongation and puncture strength was maximized. The results obtained for alginate and glycerol concentrations were 2 and 1.5%, respectively, using the dry method for tilm formation. According to the above values and FFM, for each response variables the predicted values were 0.1423 mm for thickness, 69.2% of moisture content, 29.8% for water solubility, 4.68 g mm / kPa m2 h for water vapor permeability, 220.1% for percentage elongation, and 21.7 MPa for puncture strength, reaching a composite desirability of 0.97868.

IV. Conclusion

The film forming method did not have significant effect in hue angle and lightness of edible films.

Prediction equations for responses studied are proved to be adequate to describe the experimental data on the thickness, moisture content, water solubility and water vapor permeability ($R^2 > 0.999$).

Edible films should be chosen based on their suitability for the desired and application. Therefore, it is important to

consider the components used in the formulation as well as the training in obtaining films as a decisive influence on their physical properties, directly affecting the organoleptic quality of coated food.

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