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Carbohydrate Polymers 56 (2004) 129–135
Carbohydrate Polymers
www.elsevier.com/locate/carbpol

Barrier, mechanical and optical properties of plasticized yam starch films

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Received 10 February 2003; revised 27 October 2003; accepted 22 January 2004

Available online 12 March 2004

Abstract

Yam starch films were obtained by casting. Effect of different film thicknesses (0.07, 0.09, 0.11 mm) and concentrations of glycerol (1.30, 1.65 and 2.00% w/w) and starch (3.30, 3.65 and 4.00% w/w) were evaluated. Barriers, mechanical and optical properties of films were analyzed. Films were homogeneous and transparent without insoluble particles. Water vapor and O_2 permeabilities increased with glycerol concentration. Opacity of yam starch films depended on film thickness, higher the thickness, more opaque the sample. Films with 4.00% w/w starch, 1.30% w/w glycerol and 0.11 mm thickness gave the highest puncture strength. Puncture deformation increased with glycerol content.

Keywords: Yam starch; Biofilms; Mechanical properties; Permeability

1. Introduction

Biopolymer films and coatings from polysaccharides, proteins and lipids, formulated either with one or more components have the potential to control mass transfer and thus extend food shelf life (García, Martino, & Zaritzky 1999, 2000; Parris, Coffin, Joubran, & Pessen, 1995). As they are biodegrable, biopolymers could contribute to new solutions in reducing the amount of plastic wastes. In addition, these polymers are obtained from renewable sources unlike synthetic polymers (Souza & Andrade, 2000).

The first studies about the use of starch in biodegradable food packaging were based on substituting part of the synthetic matrix by starch (below 10%), however, the main difficulties found were attributed to chemical incompatibility of starch with synthetic polymers (Griffin, 1977). Recently, many reports deal with the addition of plasticizers to pure starch-based materials to overcome film brittleness caused by high intermolecular forces (Bader & Göritz, 1994; García et al., 2000; Lourdin, Della Valle, & Colonna, 1995; Souza & Andrade, 2000). The most commonly used plasticizers used are polyols, such as sorbitol and glycerol. They avoid cracking of the film during handling and storage (Gontard, Guilbert, & Cuq, 1993) and affect gas, water vapor and solute permeabilities (Banker, 1966).

Barrier properties of potato, maize (García et al., 1999, 2000) and tapioca starch films (Souza & Andrade, 2000; Chang, Cheah, & Seow, 2000) are well characterized. Starch films are excellent oxygen barriers, due to their tightly packed, ordered hydrogen-bonded network structure and low solubility (McHugh & Krochta, 1994). On this basis, yam starch is a promising polymer for biofilm production due to their homogeneous matrix and stable structure at ambient conditions and relatively low water barrier properties (Mali, Grossmann, García, Martino, & Zaritzky, 2002).

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doi:10.1016/j.carbpol.2004.01.004

The objectives of the present work were to analyze the effects of thickness, glycerol and yam starch content on mechanical, optical and barrier properties (water vapor, oxygen and carbon dioxide) of yam starch films.

2. Materials and methods

2.1. Materials

Fresh tubers of yam (*Dioscorea alata*), with uniform size and shape, without any mechanical and pathological injuries, were obtained from a local farm (Londrina, PR, Brazil). Yam starch was extracted according to Alves, Grossmann, and Silva (1999). Amylose and amylopectin content of yam starch was determined simultaneously by Landers, Gbur, and Sharp (1991) method; ash, protein, fat and starch were determined according to the standard AOAC (1995) techniques. Glycerol (98–99% purity) was purchased from Merck (Germany) and all of reagents used in the analyses were analytical grade.

2.2. Film preparation

Yam starch films with different thicknesses (0.07, 0.09 and 0.11 mm) were prepared by casting as previously described (Mali et al., 2002). In the solution for casting glycerol concentrations were (1.30, 1.65 and 2.00% w/w) and yam starch concentrations (3.30, 3.65 and 4.00% w/w). Films, which could be easily removed from the plate, were equilibrated at 20 °C and a relative humidity (RH) of 64%, for 48 h, before being tested.

2.3. Film characterization

2.3.1. Thickness measurements

Thickness of films was determined using a manual micrometer Mitutoyo (SãoPaulo-Brazil) at 10 random positions on the films. The mean standard deviation within the film was about 5% of the average thickness.

2.3.2. Water content

Film water content was determined gravimetrically by drying small pieces in a ventilated oven model TE-394-3 (Tecnal, Piracicaba, SP, Brazil) at 105 °C overnight; and was expressed as g of water per 100 g of dried film.

2.3.3. Gas permeability

Oxygen (O_2) permeability of the films was assessed by the accumulation method in a specially designed stainless steel cell as described by García et al. (2000). O_2 concentration was measured in a gas chromatograph Shimadzu (Kyoto-Japan) with a Alltech CTRI column (Alltech Associates, Deerfield-USA). O_2 permeability of films was calculated and expressed in (cm^3 gas $m^{-1}\,s^{-1}\,Pa^{-1}$) at standard temperature (20 °C) and RH (75%). O_2 was stabilized passing through a NaCl saturated solution. All tests were conducted in duplicate.

2.3.4. Water vapor permeability (WVP)

WVP tests were conducted using ASTM (1996) method E96 with some modifications. Each film sample was sealed over a circular opening of 0.00181 m^2 in a permeation cell that was stored at 25 °C in a desiccator. To maintain 75% RH gradient across the film, anhydrous calcium chloride (0% RH) was placed inside the cell and a sodium chloride saturated solution (75% RH) was used in the desiccator. The RH inside the cell was always lower than the outside, and water vapor transport was determined from the weight gain of the permeation cell. After steady state conditions were reached (about 2 h), eight weight measurements were made over 24 h. Changes in the weight of the cell were recorded to the nearest 0.0001 g and plotted as a function of time. The slope of each line was calculated by linear regression ($r^2 > 0.99$) and the water vapor transmission rate (WVTR) was calculated from the slope of the straight line (g/s) divided by the transfer area (m^2). After the permeation tests, film thickness was measured and WVP ($g\,Pa^{-1}\,s^{-1}\,m^{-1}$) was calculated as $WVP = [WVTR/S(R_1 - R_2)d]$; where S is the saturation vapor pressure of water (Pa) at the test temperature (25 °C), R_1, the RH in the desiccator, R_2, the RH in the permeation cell and d is the film thickness (m). Under these conditions, the driving force $[S(R_1 - R_2)]$ was 1753.55 Pa. All tests were conducted in duplicate.

2.3.5. Opacity

Opacity of the films was determined using a BSI standard procedure (1968) modified by Gontard, Guilbert, and Cuq (1992). Film sample were cut to 1 × 3 cm and placed on the internal side of a spectrophotometer cell (GBC Cintra 20, Vitória-Austrália) to record the absorbance spectrum between 400 and 800 nm. Film opacity was defined as the area under the curve and expressed as Absorbance Units × nanometers (AU nm). All tests were conducted in duplicate.

2.3.6. Mechanical properties

Puncture tests were made to determine strength (N) and deformation (mm) using a TA.TX2i Stable Micro Systems texture analyzer (Surrey-England). Samples with diameters of 40 mm were fixed on the plate of the equipment over a hole of 20 mm diameter with the help of adhesive tape (3M Scotch, Brazil). A cylindrical probe of 5 mm diameter was moved perpendicularly to the film surface at a constant speed of 1 mm/s until the probe passed through the film. Force-deformation curves were recorded. Force and deformation were recorded at the rupture point. For each test eight samples were analyzed.

2.3.7. Statistical design

A full factorial design (2^3) was adopted to determine the influence of three independent variables, at two levels each, on film properties. The complete design consisted of 10

Table 1
Responses of dependent variables to the film-forming conditions

Run	Independent variables[a] (coded and real values)			WC	Dependent variables[b]				
	x_1	x_2	x_3		Y_1	Y_2	Y_3	Y_4	Y_5
1	−1 (0.07)	−1 (1.30)	−1 (3.30)	23.53	2.92	0.989	85.0	8.02	3.44
2	−1 (0.07)	−1 (1.30)	1 (4.00)	24.08	2.86	1.150	95.0	9.84	3.48
3	−1 (0.07)	1 (2.00)	−1 (3.30)	25.71	3.89	1.071	98.4	6.03	4.78
4	−1 (0.07)	1 (2.00)	1 (4.00)	23.79	3.64	1.395	88.7	9.61	4.20
5	1 (0.11)	−1 (1.30)	−1 (3.30)	27.42	2.17	1.298	96.4	11.46	4.20
6	1 (0.11)	−1 (1.30)	1 (4.00)	27.19	2.78	1.685	111.2	15.96	3.95
7	1 (0.11)	1 (2.00)	−1 (3.30)	25.72	3.03	1.550	101.7	8.73	4.38
8	1 (0.11)	1 (2.00)	1 (4.00)	25.31	3.90	1.810	97.2	14.89	4.61
9	0 (0.09)	0 (1.65)	0 (3.65)	25.83	3.75	1.270	94.3	10.62	4.43
10	0 (0.09)	0 (1.65)	0 (3.65)	25.42	3.39	1.375	87.7	10.17	4.48

[a] x_1, thickness (mm); x_2, glycerol concentration (%) and x_3, starch concentration (%).
[b] WC, Water content (g water/100 g dried film), Y_1, O_2 permeability × 10^{10} ($cm^3\ m^{-1}\ s^{-1}\ Pa^{-1}$); Y_2, water vapor permeability × 10^{10} ($g\ m^{-1}\ s^{-1}\ Pa^{-1}$); Y_3, opacity (Au nm); Y_4, puncture strength (N) and Y_5, puncture deformation (mm).

experimental points including two replicates at the central point, which were included to estimate the pure error of the analysis and to predict the lack of fit of the models. The three independent variables were film thickness (x_1), glycerol concentration (x_2) and starch concentration (x_3) in the casting solution. Variable levels were chosen from preliminary studies. The real levels of independent variables were 0.07, 0.09 and 0.11 mm for thickness (x_1), 1.30, 1.65 and 2.00% w/w for glycerol concentration (x_2) and 3.30, 3.65 and 4.00% w/w for yam starch concentration (x_3). The real levels of independent variables were coded as −1, 0 and 1 (Table 1). The responses under observation were O_2 permeability (Y_1), WVP (Y_2), opacity (Y_3), puncture strength (Y_4) and puncture deformation (Y_5).

Experimental data were analyzed by SAS RSREG procedure to fit polynomial models for each dependent variable and stepwise procedure was used to simplify the models (SAS Institute, 1995). Three-dimensional surface plots were generated from obtained models by Statistica Software (Oklahoma, USA).

3. Results and discussion

3.1. Chemical composition of yam starch

On dry basis, the chemical composition of yam starch was: ash (0.17 ± 0.01%), protein (0.20 ± 0.01%), lipids (0.27 ± 0.02%) and starch (98.30 ± 0.05%). The amylose and amylopectin contents were 30 and 70%, respectively. This amylose content is important for the film forming capacity of the starch; it was higher than the values obtained by other authors (Ciacco, 1978; Emiola & Delarosa, 1981) and similar to those obtained by Alves et al. (1999). The differences could be explained by the different plantation conditions of yam tubers and by the analytical procedure.

Table 2
Regression coefficients for dependent variables and analysis of variance of the polynomial models

Coefficients		WC	Y_1	Y_2	Y_3	Y_4	Y_5
Linear	β_o	25.40	3.23	1.35	95.96	10.53	4.19
	β_1	1.07**	−0.17	0.22***	4.93**	2.19***	0.16
	β_2	−0.21	0.47**	0.09***	−0.20	0.75***	0.36**
	β_3	−0.25	0.15	0.14***	1.33	2.01***	
Interaction	β_{12}						
	β_{13}					0.66***	
	β_{23}				−4.88**	0.43**	
Coefficient of determination (R^2)		0.85	0.75	0.90	0.80	0.99	0.70
Model significance (p)		−	0.0362	0.0001	0.0725	0.0001	0.0184
Coefficient of variation (%)		6.78	6.43	6.55	3.45	2.13	4.82
Lack of fit significance (p)		0.32	0.64	0.40	0.98	0.93	0.81

$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + e$, x_1 = thickness, x_2 = glycerol concentration and x_3 = starch concentration. *, ** and ***, significant at $p \le 0.1, p \le 0.05, p \le 0.01$, respectively. WC, water content (g water/100 g of dried film), Y_1, O_2 permeability × 10^{10} ($cm^3\ m^{-1}\ s^{-1}\ Pa^{-1}$), Y_2, water vapor permeability × 10^{10} ($g\ m^{-1}\ s^{-1}\ Pa^{-1}$), Y_3, opacity (Au nm) Y_4, puncture strength (N), Y_5, puncture deformation (mm).

3.2. Water content

Water content of yam starch films is shown in Table 1, values ranged from 23.63 to 27.72 (g water/100 g dried film). These data agreed with those of Chang et al. (2000) who worked with tapioca starch films; they stressed that water below 27% moisture was nonfreezable and this was confirmed by DSC studies.

Effects of thickness, glycerol and starch concentrations on water content were analyzed by linear regression. ANOVA (Table 2) showed that only thickness was a significant factor ($p < 0.05$); water content increased with thickness (Table 1). This occurred probably because the higher thicknesses resulted in more hydrophilic groups susceptible to interact with water. The glycerol content did not influence the water content possibly because it was varied over a relatively narrow range.

3.3. Statistical and response surface analysis

Film-forming conditions and film properties are shown in Table 1. Table 2 summarizes the ANOVA results for each dependent variable response.

According to ANOVA (Table 2), the models for WVP (Y_2) and puncture strength (Y_4) were significant at 1% level ($p \leq 0.01$), for O_2 permeability (Y_1) and deformation (Y_5) were significant at 5% level ($p \leq 0.05$) and for opacity (Y_3) at 10% level ($p \leq 0.10$). O_2 permeability (Y_1) and puncture deformation (Y_5) models had $R^2 < 0.80$, these low values could be attributed to the simplification of the models. All models showed a coefficient of variation below 6.55% and no significant lack of fit. Thus, the models agreed with experimental data and are useful for studying the effect of film forming conditions on film properties.

3.4. Gas permeability

According Table 2, O_2 permeability was exclusively influenced by the linear effect of glycerol (x_2); increasing glycerol content increased gas permeability (Fig. 1). Our results agreed with those of Banker (1966) and McHugh and Krochta (1994), who stressed that plasticizers such as glycerol generally increase gas and water vapor permeabilities of hydrophilic films. Glycerol is a relative small hydrophilic molecule and has a similar chemical structure to glucose, thus glycerol can easily interact with starch chains reducing packing between chains. Thus, the polymer chain movements could be facilitated, resulting in an increase of water vapor and gas permeation through the film matrix.

3.5. Water vapor permeability (WVP)

Since a main function of a food packaging is often to avoid or at least to decrease moisture transfer between the food and the surrounding atmosphere, or between two components of a heterogeneous food product, WVP should be as low as possible (Gontard et al., 1992).

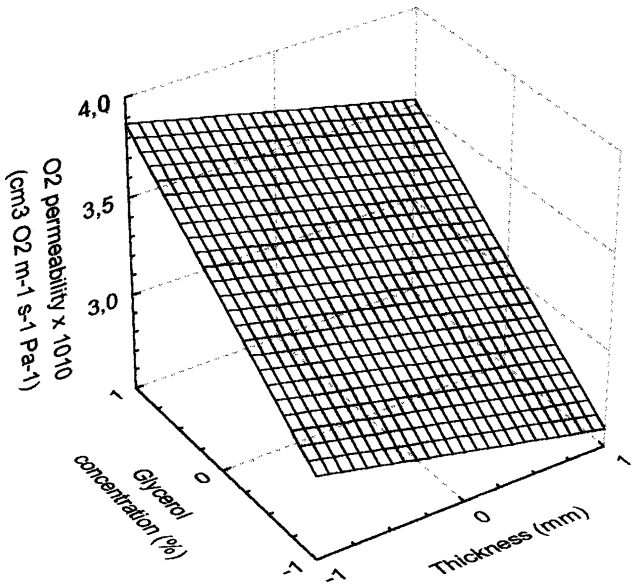

Fig. 1. Response surface for the effects of thickness and glycerol concentration on O_2 permeability, at a fixed starch concentration of 3.65%.

WVP of yam starch films was influenced by the linear effects of thickness (x_1), glycerol concentration (x_2) and starch concentration (x_3), (Table 2); WVP increased when these three variables increased (Fig. 2 a,b).

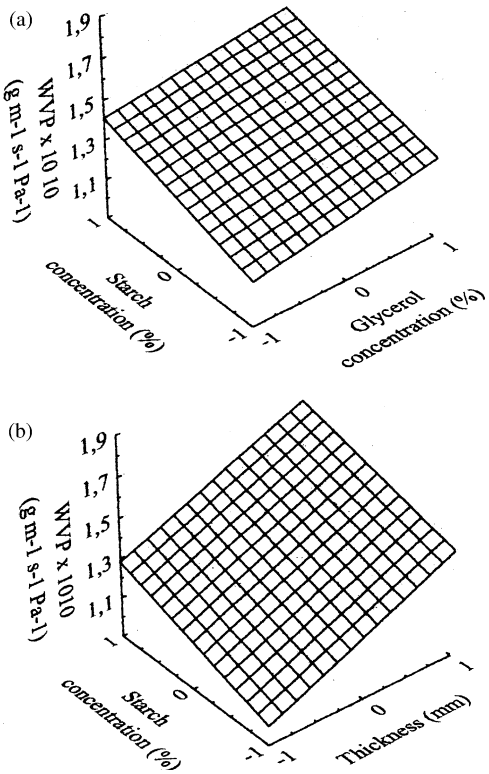

Fig. 2. Water vapor permeability: (a) effects of glycerol and starch concentrations at a fixed thickness of 0.09 mm and (b) effects of thickness and starch concentration at a fixed glycerol concentration of 1.65%.

Hydrophilic films exhibiting increased WVP with film thicknesses were also reported by other researches as Banker (1966); Swartzberg (1986); Hagenmaier and Shaw (1990); McHugh, Avena-Bustillos, and Krochta (1993); Cuq, Gontard, Cuq, and Guilbert (1996); Sobral (2000). Several explanations have been provided for such anomalous thickness effect. According to Swartzberg (1986), biofilms containing hydrophilic components, such as polysaccharides, have a extremely complex transport through them, due to non-linear water sorption isotherms and water-content-dependent diffusivities. Thus, water vapor flux through hydrophilic films varies non-linearly with water vapor pressure gradient. Park and Chinnan (1995) reported that this anomalous effect occurred because the swelling of hydrophilic matrix results in different structural changes in films with different thicknesses.

WVP values increased with glycerol content (Fig. 2a). With regard to the plasticizing effect of glycerol, a similar trend as for O_2 permeability was observed for WVP. Similar results have been reported on protein films by Gontard et al. (1993). This behavior could be related to structural modifications of the starch network that might become less dense, in combination with the hydrophilic character of glycerol, which is favorable to adsorption and desorption of water molecules. Moreover, in previous studię, water sorption isotherms showed that above 43% RH the equilibrium water content of starch films with glycerol was higher than those of non-plasticized films (Mali et al., 2002; Myllärinen, Partanen, Seppälä, & Forsell, 2002).

Yam starch films exhibited increased WVP values at increased starch concentration (Fig. 2 a,b), what could be related with a higher number of free hydroxyl groups, that might enhanced interactions with water, favoring water vapor transmission through the films. According to Cuq, Gontard, and Guilbert (1998) most free hydrophilic groups of proteins favor sorption and water vapor transfer, rather than hydrophobic gas transfer like that of CO_2 and O_2. This observation agreed with our data; starch concentration was a significant factor only for WVP but not for O_2 permeability (Tables 1 and 2).

With regard to synthetic polymers, yam starch films have WVP values higher than these of low-density polyethylene (LPDE) (0.0036×10^{-10} g m^{-1}s^{-1} Pa^{-1}) (Shellhammer & Krochta, 1997). However, yam starch film permeabilities were lower than those of other biodegradable films such as wheat gluten plasticized with glycerol (7.00×10^{-10} g m^{-1}s^{-1} Pa^{-1}), amylose (3.80×10^{-10} g m^{-1}s^{-1} Pa^{-1}) and hydroxypropyl- methylcellulose with plasticizer and oil (1.90×10^{-10} g m^{-1}s^{-1} Pa^{-1}) (Gennadios, Weller, & Gooding, 1994), and slightly higher than those of cellophane (0.84×10^{-10} g m^{-1}s^{-1} Pa^{-1}) (Shellhammer & Krochta, 1997) and methyl cellulose films (0.500×10^{-10} g m^{-1}s^{-1} Pa^{-1}) (Turhan & Sahbaz, 2003).

3.6. Visual appearance and opacity

All formulations gave easy handling, homogeneous and transparent films without any insoluble particles.

Opacity is a property of prime importance if the film is to be used as a food coating or as a food packaging (Gontard et al., 1992). Low relative opacity values indicated a transparent film. Based on the significant regression coefficients (Table 2), film opacity was influenced by the linear effect of thickness (x_1) and by interaction of glycerol content with starch concentration (x_2x_3); an increase in film thickness caused an increase in film opacity (Fig. 3). Experimental opacities of yam starch films ranged between 85.0 and 111.2 Au nm, showing a better performance as compared to those of wheat gluten films, measured by the same method, which showed an opacity of 250.4 Au nm (Gontard, 1991).

3.7. Mechanical properties

The desired property of a food packaging material depends on the application. In general, a food packaging may be an undeformable material to provide structural integrity or reinforce food structure, or a deformable film for other applications (Gontard et al., 1992).

According to ANOVA (Table 2), puncture strength was influenced by the linear effects of thickness (x_1), glycerol concentration (x_2) and yam starch concentration (x_3) and by the interactions of the starch concentration with thickness (x_3x_1) and with glycerol concentration (x_3x_2). The response surfaces (Fig. 4a,b) showed that higher thickness and higher starch concentrations combined with lower glycerol contents induced formation of resistant films with higher puncture strengths.

Puncture strength was enhanced with increasing starch content; during drying of film-forming solutions, water

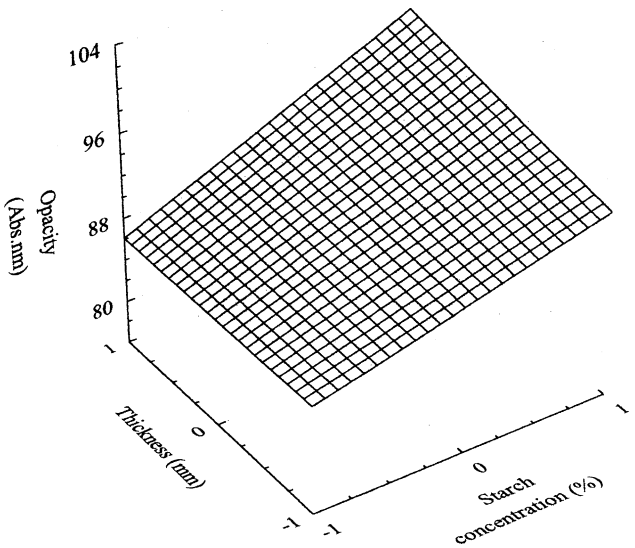

Fig. 3. Response surface for the effects of thickness and starch concentration on opacity, at a fixed glycerol concentration of 1.65%.

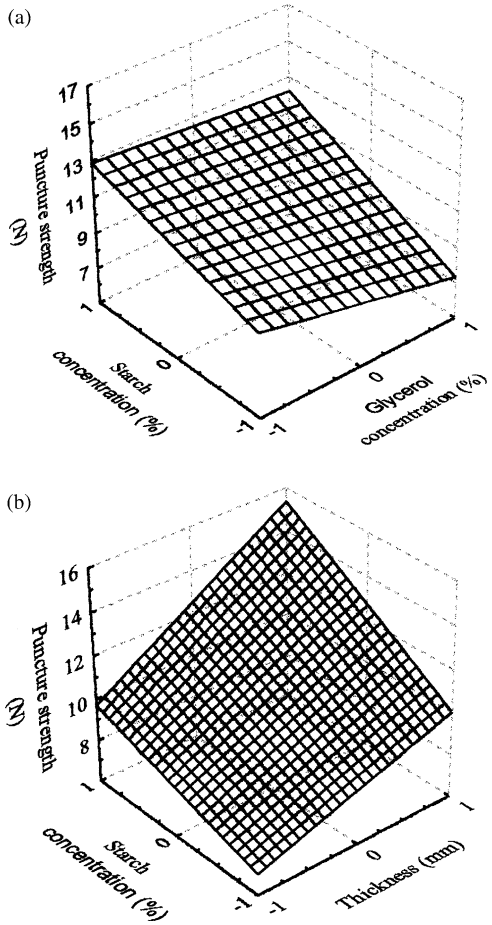

Fig. 4. Puncture strength: (a) effects of glycerol and starch concentrations at a fixed thickness of 0.09 mm and (b) effects of thickness and starch concentration at a fixed glycerol concentration of 1.65%.

evaporates, allowing the formation of starch network, and during this stage, the proximity of starch chains favored by higher starch contents could facilitate the formation of a more dense matrix. Similar results have been reported by Gontard et al. (1992), which showed that puncture strength of wheat gluten films was improved as gluten concentration increased.

With regard to thickness, puncture strength increased with film thickness. To obtain films by casting with higher thicknesses, either higher mass of the same solution or a higher concentration of film-forming solution has to be used. This effect was analyzed on protein films (Sobral, 2000).

Puncture deformation had a linear dependence on glycerol concentration (x_2), (Table 2). Fig. 5 shows deformation increase with glycerol content with thickness having little effect.

Puncture strength varied between 6.03 and 15.96N, while deformation ranged between 3.44 and 4.78 mm. Gontard et al. (1992) working with plasticized whey protein films, reported puncture strength values between 0.22 and 3.34 N and deformations between 4.8 and 17.6 mm. Monterrey and Sobral (1999) reported puncture strength ranging between

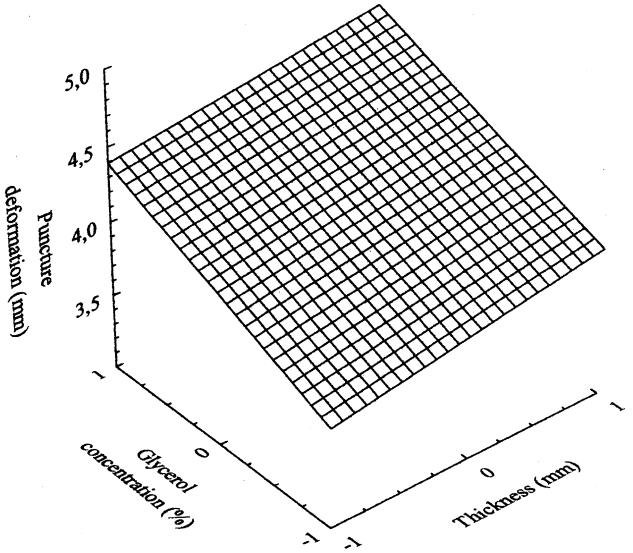

Fig. 5. Response surface for the effect of thickness and glycerol concentration on puncture deformation.

2.94 and 6.67N and deformations between 2.71 and 7.50 mm for myofibrillar protein films.

The physical state of films strongly affects mechanical and barrier properties. The glass transition temperature (T_g) is a good indicator of the physical state of partially crystalline materials. In a previous work we determined the glass transition temperature of yam starch films with the same composition and we found that films were in the rubbery state at ambient temperature ($T_g < 25$ °C) furthermore (T_g) decreased with glycerol concentration (Mali et al., 2002). The lowest (T_g) (8.81 °C) corresponded to films with the highest glycerol/starch ratio (runs 3 and 7, Table 1). Thus, these films showed the highest deformation values (Fig. 5), evidencing the structural modifications of starch network when glycerol was incorporated. The matrix of the film becomes less dense with glycerol addition and under stress, movement of polymer chains were facilitated. A similar trend was reported by other authors working on hydrophillic films (Cuq, Aymard, Cuq, & Guilbert, 1995; Parris et al., 1995; Monterrey & Sobral, 1999).

4. Conclusions

Glycerol concentration and thickness had a marked influence on various film properties. The plasticizing effect of glycerol resulted in, WVP, O_2 permeability and puncture deformation increasing with glycerol concentration while puncture strength decreased.

Response surface methodology proved to be an effective tool for this type of study because of the complexity of film forming conditions involving interrelated variables.

The selection of the optimum film-forming condition depends on the specific use of the film, application techniques and other considerations. For example, if the film were to be used to protect products from handling

abuse the prime property to optimize would be mechanical. Within the range of the tested formulations, high starch concentration (4.00%), high thickness (0.11 mm) and low glycerol concentration (1.30%) would be recommended for films with good barrier properties.

Acknowledgements

The authors wish to thanks the financial support provided by CAPES (ProDoc) and CNPq (Brazil), SECyT (Argentina) and CYTED (Program XI).

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