**FOOD HYDROCOLLOIDS**

# Mechanical and thermal properties of yam starch films

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## Abstract

Yam starch films were prepared by casting using glycerol as plasticizer. The effects of different film thicknesses (0.07, 0.09, 0.11 mm), glycerol (1.30, 1.65 and 2.00 g/100 g of filmogenic solution) and starch (3.30, 3.65 and 4.00 g/100 g of filmogenic solution) concentrations were evaluated in mechanical properties of these films. Films with high puncture strengths were obtained at higher thickness, higher starch concentration and lower glycerol concentration. Puncture deformation increased with glycerol content. Tensile tests showed that with increasing of glycerol concentration, the films became more flexible, with higher strain at break values and lower stress and Young's Modulus. Glass transition temperatures ($T_g$) of films were affected by glycerol and starch concentration; films formulated with high yam starch concentrations showed higher $T_g$ values and $T_g$ of film formulated without plasticizer (control sample) were higher than those of plasticized films.
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## 1. Introduction

Since the 1970s, there has been a renewed interest in biodegradable plastics made from renewable and natural polymers. Among the natural polymers, starch has been considered as one of the most promising candidates for future materials because of attractive combination of price, abundance and thermoplastic behavior.

Starch is a raw material with thermoplastic properties upon disruption of its molecular structure (Tharanathan, 1995; Tharanathan & Saroja, 2001); besides is a complex homopolymer of α-D-glucose units and is found mainly in two structurally and functionally distinct forms—amylose and amylopectin. Amylose is a linear or sparsely branched polymer and amylopectin is a highly branched polymer (Van Soest & Vliegenthart, 1997; García,

Martino, & Zaritzky, 1999; 2000). Increasing amylose content in the starches gives stronger films. Branched structure of amylopectin generally leads to films with poor mechanical properties (Tharanathan, 2003). 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 barrier, 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 because it contains about 30% of amylose, and this is important for film production because amylose is responsible for the film-forming capacity of starches. Yam starch films were described as films with a homogeneous matrix, with stable structure at ambient conditions and a poor water vapor barrier compared with synthetic materials, which could be promising in the post-harvest conservation of fruits and vegetables (Mali, Grossmann, García, Martino, & Zaritzky, 2002; Mali & Grossmann, 2003).

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To overcome brittleness inherent of starch films, the incorporation of a plasticizer is required. Plasticizers reduce intermolecular forces and increase the mobility of polymer chains, decreasing the glass transition temperature of these materials and improve their flexibility. They must be compatible with the film-forming polymers; hydrophilic compounds, such as polyols (glycerol and sorbitol) are commonly used in starch films (García et al., 1999, 2000)

According to Van Soest and Vliegenthart (1997), plasticized starch films are classified as a function of plasticizer content: a glassy behavior at low plasticizer content and, at higher plasticizer content, a rubbery behavior proceeded by a glass-to-rubber transition. The glass transition temperature ($T_g$) characterizes the glass-to-rubber transition; glassy materials are hard and stiff but can be brittle and rubbery materials are more flexible.

Thus, the objectives of the present work were to analyze the effects of thickness, glycerol and yam starch content on mechanical properties of yam starch films and its relationships with $T_g$.

## 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 et al. (1999). Amylose and amylopectin content of yam starch were determined simultaneously by Landers, Gbur and Sharp (1991) method; ash, moisture, protein, fat and starch were determined according to the standard AOAC (1995) techniques. Glycerol (98–99% purity) was purchase from Merck (Germany) and all of reagents used in the analyses had analytical grade.

### 2.2. Film preparation

Yam starch films were prepared as previously described (Mali et al., 2002), with different thicknesses, glycerol and yam starch concentrations. For each experiment, the quantity of starch suspension poured onto the plate was calculated to obtain a constant weight of dried matter of approximately 8.30, 11.00 and 12.25 mg/cm$^2$ for 0.07, 0.09 and 0.11 mm films, respectively. The obtained translucent films could be easily removed from the plate and were equilibrated at 20 °C and a relative humidity (RH) of 64%, for 48 h, before being tested. 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. Film characterization

#### 2.3.1. Thickness measurements

Thickness of films was determined using a manual micrometer Mitutoyo with an accuracy of ±1 μm (São Paulo, 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. Mechanical properties

*2.3.2.1. Puncture tests.* Puncture tests were made to determine force ($N$) 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 with a hole of 20 mm diameter with help of a 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. At rupture point force and deformation were determined. For each test eight samples were analyzed.

*2.3.2.2. Tensile tests.* The tensile properties were studied using an Instron 1141 (Instron Ltd, Canton, USA) in accordance with ASTM D-882-91 (1996). Five sample strips (40 × 150 mm$^2$) of each formulation were cut and clamped between pneumatic grips. Force ($N$) and deformation (mm) were recorded during extension at 50 mm min$^{-1}$ and with an initial distance between the grips of 100 mm. The parameters determined were: stress at break (MPa), strain at break (%) and Young's modulus (MPa). Stress at break and strain at break are often also referred in literature as tensile strength and elongation.

#### 2.3.3. Thermal properties—determination of glass-transition temperature ($T_g$)

The glass transition temperature ($T_g$) of yam starch films were analyzed by thermomechanical analysis (TMA); the $T_g$ was analyzed only as a function of glycerol and starch content, the different thicknesses were not considered because did not influenced this phenomena in preliminary studies.

Film samples were tested in Polymer Laboratories TMA 500 (Rheometric Scientific, Surrey, UK) working under PLus-V5.40 software with a liquid nitrogen Dewar attachment. Samples of 20–40 mg and 2–3 mm in diameter were placed in open aluminum pans. A penetration standard probe of quartz was used and the applied force was 980 mN (10 g). Samples were heated from −80 to 90 °C with 10 °C/min heating rate. The temperature of glass transition was determined by the change in the slope of the obtained TMA curves (dimension change versus temperature). All measurements were performed in duplicate. A control sample without glycerol was tested to compare with the plasticized films.

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

| Run | Independent variables[a] (coded and real values) | | | Deapendent variables[b] | | | | | |
|-----|------|------|------|------|------|------|------|------|------|
| | $x_1$ | $x_2$ | $x_3$ | $y_1$ | $y_2$ | $y_3$ | $y_4$ | $y_5$ | WC |
| 1 | −1 (0.07) | −1 (1.30) | −1 (3.30) | 8.02 | 3.44 | 7.84 | 10.3 | 201 | 23 ± 2 |
| 2 | −1 (0.07) | −1 (1.30) | 1 (4.00) | 9.84 | 3.48 | 5.63 | 28.5 | 121 | 24 ± 2 |
| 3 | −1 (0.07) | 1 (2.00) | −1 (3.30) | 6.03 | 4.78 | 3.82 | 30.0 | 46 | 26 ± 3 |
| 4 | −1 (0.07) | 1 (2.00) | 1 (4.00) | 9.61 | 4.20 | 4.18 | 37.0 | 48 | 24 ± 2 |
| 5 | 1 (0.11) | −1 (1.30) | −1 (3.30) | 11.46 | 4.20 | 3.41 | 35.7 | 46 | 27 ± 4 |
| 6 | 1 (0.11) | −1 (1.30) | 1 (4.00) | 15.96 | 3.95 | 4.70 | 47.0 | 89 | 27 ± 3 |
| 7 | 1 (0.11) | 1 (2.00) | −1 (3.30) | 8.73 | 4.38 | 2.84 | 34.5 | 28 | 26 ± 2 |
| 8 | 1 (0.11) | 1 (2.00) | 1 (4.00) | 14.89 | 4.61 | 5.00 | 41.0 | 111 | 25 ± 3 |
| 9 | 0 (0.09) | 0 (1.65) | 0 (3.65) | 10.62 | 4.43 | 4.17 | 38.0 | 51 | 26 ± 4 |
| 10 | 0 (0.09) | 0 (1.65) | 0 (3.65) | 10.17 | 4.48 | 4.07 | 36.7 | 45 | 25 ± 2 |

[a] $x_1$ =thickness (mm), $x_2$ =glycerol concentration (%) and $x_3$ =starch concentration (%).

[b] $Y_1$ =puncture force (N), $Y_2$ =puncture deformation (mm), $Y_3$ =stress at break (MPa), $Y_4$ =strain at break (%), $Y_5$ =Young's Modulus (MPa) and WC = water content (g water/100 g dried film).

## 2.3.4. Statistical design

A full factorial design ($2^3$) was adopted to determine the influence of the three independent variables, at two levels each, on film properties. The complete design consisted of 10 experimental points including two replications at center 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 ($x_3$) on filmogenic solution and the levels were chosen from preliminary studies (Mali et al., 2002). The real levels of independent variables were 0.07, 0.09 and 0.11 mm for film thickness ($x_1$), 1.30, 1.65 and 2.00 g/100 g of filmogenic solution for glycerol concentration ($x_2$) and 3.30, 3.65 and 4.00 g/100 g of filmogenic solution 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: puncture force ($Y_1$), puncture deformation ($Y_2$), stress at break ($Y_3$), strain at break ($Y_4$) and Young's modulus ($Y_5$).

Experimental data were analyzed to fit polynomial models to response variables by SAS RSREG procedure and stepwise procedure was employed to simplify the models (SAS, 1995). Three-dimensional surface plots were generate from 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 ($0.17 \pm 0.01\%$), protein ($0.20 \pm 0.01\%$), lipids ($0.27 \pm 0.02\%$) and starch ($98.30 \pm 0.05\%$). The amylose and amylopectin contents were 30 and 70%, respectively. This amylose content is relevant for the film forming capacity of the starch and was higher than the values obtained by other authors (Ciacco, 1978; Emiola & Delarosa, 1981)

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

| Coefficients | | $Y_1$ | $Y_2$ | $Y_3$ | $Y_4$` | $Y_5$ |
|-----|-----|-----|-----|-----|-----|-----|
| | $\beta_o$ | 10.53 | 4.19 | 4.54 | 33.9 | 48 |
| Linear | $\beta_1$ | 2.19*** | 0.16 | −0.65 | 6.6*** | −18** |
| | $\beta_2$ | −0.75*** | 0.36** | −0.68* | 2.6 | −28** |
| | $\beta_3$ | 2.01*** | | | 5.4** | 6 |
| Interaction | $\beta_{12}$ | | | 0.62 | −4.4** | 29** |
| | $\beta_{13}$ | 0.66*** | | | | 25** |
| | $\beta_{23}$ | 0.43** | | | | 15* |
| Coefficient of determination ($R^2$) | | 0.99 | 0.70 | 0.65 | 0.92 | 0.96 |
| Model significance (p) | | 0.0001 | 0.0184 | 0.0818 | 0.074 | 0.0337 |
| Coefficient of variation (%) | | 2.13 | 4.82 | 14.50 | 5.10 | 14.14 |
| Lack of fit significance (p) | | 0.93 | 0.81 | 0.06 | 0.25 | 0.17 |

$Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + + \beta_{12} x_1 x_2 + \beta_{13} x_1 x_3 + \beta_{23} x_2 x_3 + e$; $x_1$ =thickness, $x_2$ =glycerol concentration and $x_3$ =starch concentration. *, ** and *** = significant at $P \leq 0.1, P \leq 0.05, P \leq 0.01$, respectively; $Y_1$ =puncture force (N), $Y_2$ =puncture deformation (mm), $Y_3$ =stress at break (MPa), $Y_4$ =strain at break (%) and $Y_5$ =Young's Modulus (MPa).

being 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 methods used in this analysis.

## 3.2. Statistical analysis and response surfaces

Film-forming conditions and corresponding film properties data are shown in Tables 1 and 2 summarizes results of the analysis of variance (ANOVA) for each of the responses. The models for puncture force ($Y_1$) were significant at 1% of significance ($P \leq 0.01$), for puncture deformation ($Y_2$) and Young's Modulus ($Y_5$) were significant at 5% ($P \leq 0.05$) and for stress at break ($Y_3$) and strain at break ($Y_4$) at 10% ($P \leq 0.10$). Puncture deformation ($Y_2$) and stress at break ($Y_3$) models presented $R^2 < 0.80$, and this could be attributed to the simplification of the models. The coefficients of variation of the models ranged from 2.13 to 14.50% and lack of fit was not significant for any model. Thus, the models agreed with experimental data and appeared to be adequate to study the effect of film forming conditions on film properties.

## 3.3. Mechanical properties

According to ANOVA (Table 2), puncture force was influenced by effects of thickness ($x_1$), glycerol concentration ($x_2$) and yam starch concentration ($x_3$) and by interactions of the starch concentration with thickness ($x_3 x_1$) and with glycerol concentration ($x_3 x_2$). The response surfaces (Fig. 1a and b) showed that higher starch concentrations, combined with lower glycerol contents or higher thicknesses, induced formation of films with higher puncture force.

Puncture force was enhanced with increasing starch content; during drying of film-forming solutions, water evaporates, allowing the formation of starch network, and during this stage, the proximity of starch chains induced by higher starch contents could facilitate the formation of a matrix with more starch content per film area. Similar results have been reported by Gontard, Guilbert, and Cuq (1992), which showed that puncture force of wheat gluten films was improved as gluten concentration increased.

The effect of film thickness increasing puncture force was demonstrated by other authors studying protein films (Sobral, 2000). 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 employed, leading more resistant films.

The effect of plasticizer on reduction of puncture force is known and its explanation is found in literature (Parris, Coffin, Joubran, & Pessen, 1995; Sobral, Menegalli, Hubinger, & Roques, 2001; Mali et al., 2002). Sobral et al. (2001) observed a decrease in puncture force of bovine hide and pigskin gelatin films from 16.0 to 8.20 N and from 16.2 to 9.0 N, respectively,
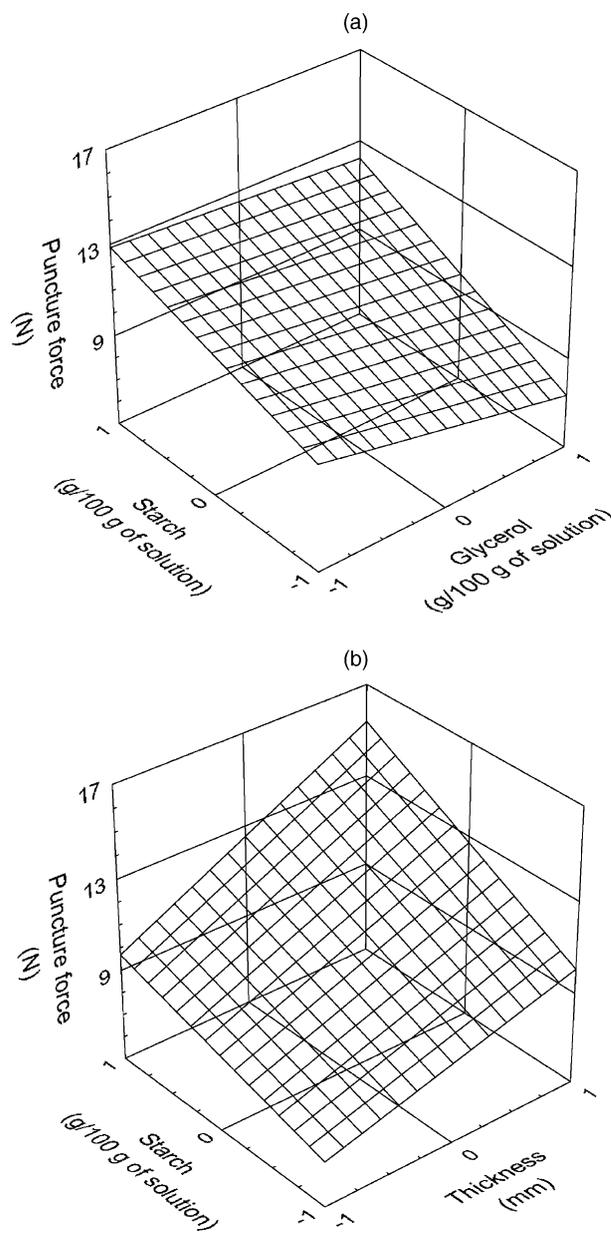


Fig. 1. Puncture force of yam starch films: (a) effects of glycerol and starch concentration at a fixed thickness of 0.09 mm and (b) effects of thickness and starch concentration at a fixed glycerol concentration of 1.65%.

with increasing of sorbitol content from 15 to 65 g/100 g gelatin.

Puncture deformation was exclusively affected by effect of glycerol concentration ($x_2$), (Table 2). Fig. 2 shows deformation increase with glycerol and the negligible effect of thickness. Gontard et al. (1992), working with plasticized wheat gluten films, found puncture deformation values ranged from 4.8 to 17.6 mm with increasing of plasticizer content. Sobral et al. (2001) observed an increase in puncture deformation of gelatin films from 1.2 to 5.3% with increasing of sorbitol content from 15 to 65 g/100 g gelatin.

The increase of puncture deformation and the decrease of puncture force with increasing glycerol content in
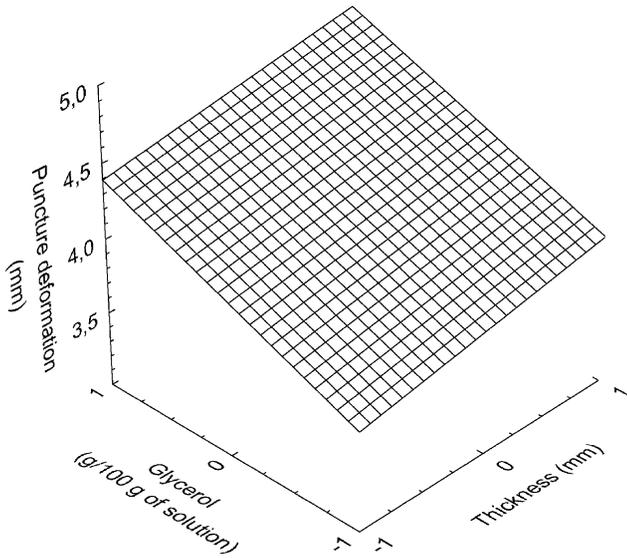
Fig. 2. Response surface for the effects of thickness and glycerol concentration on puncture deformation of yam starch films.

hydrophilic films have been reported previously (Cuq, Aymard, Cuq, & Guilbert, 1995; Parris et al., 1995; Monterrey & Sobral, 1999, Sobral et al., 2001). This behavior could be related to the structural modifications of starch network when glycerol was incorporated, the matrix of the film become less dense and under stress movements of polymer chains were facilitated.

The stress at break in yam starch films was exclusively affected by effect of glycerol concentration ($x_2$), (Table 2). Fig. 3 shows tensile stress decrease with glycerol and the negligible effect of thickness; this occurred because when

glycerol was incorporated in a starch network, direct interactions and the proximity between starch chains were reduced, thus under tensile forces, movements of starch chains were facilitated on films plasticized with glycerol.

Strain at break (%) was influenced by effect of thickness ($x_1$) and starch concentration ($x_3$) and by interaction between thickness and glycerol concentration ($x_1x_2$). As seen in Fig. 4a, strain at break (%) was higher at higher thicknesses and starch concentrations. The interaction



(a)

(b)
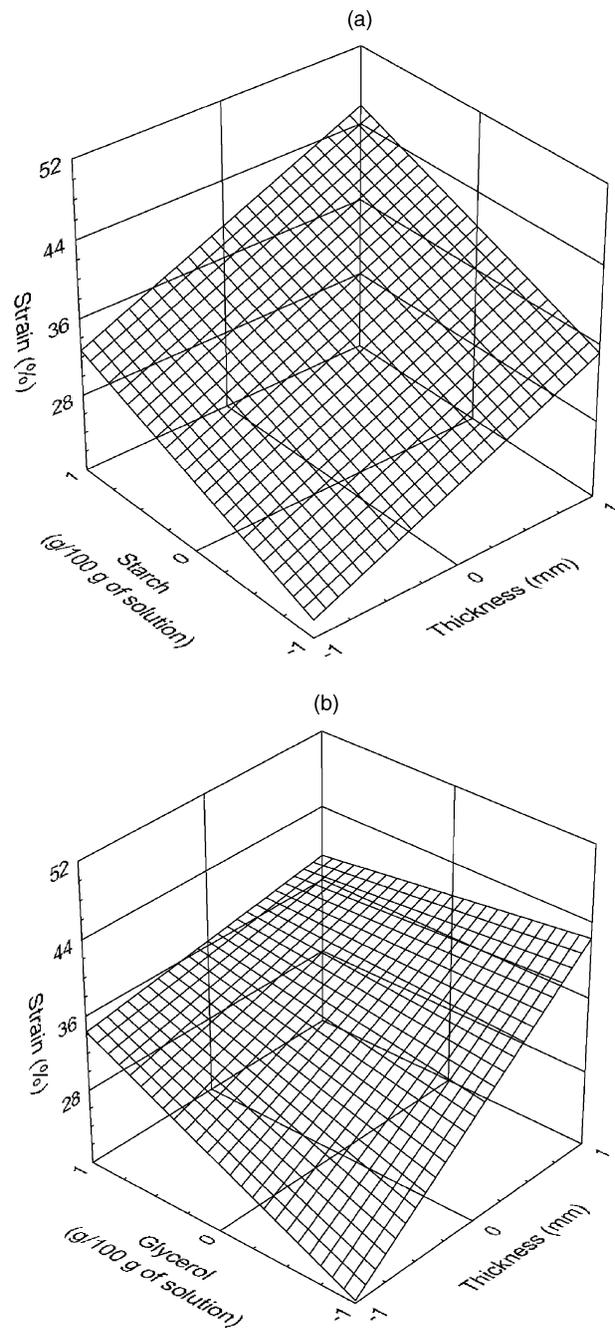
Fig. 4. Strain at break of yam starch films: (a) effects of thickness and starch concentration at a fixed glycerol concentration of 1.65% mm and (b) effects of thickness and glycerol concentration at a fixed starch concentration of 3.65%.



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

Table 3
Tensile properties of yam starch films compared to other hydrophilic films and synthetic films

| Polymer | Stress at break (Mpa) | Strain at break (%) | Comments |
|---|---|---|---|
| Yam starch[a] | 7.84 | 10.3 | Present study—Starch:glycerol = 3.3:1.3, film tested at 25 °C and 64% RH. Thickness of 0.07 mm. |
| Yam starch[b] | 4.70 | 47.0 | Present study—Starch:glycerol = 4.0:1.3, film tested at 25 °C and 64% RH. Thickness of 0.11 mm. |
| Yam starch[c] | 2.84 | 34.5 | Present study—Starch:glycerol = 3.3:2.0, film tested at 25 °C and 64% RH. Thickness of 0.11 mm. |
| Amylose | – | 31.0 | Rindlav-Westling, Stading, Hermansson & Gatenholm (1998). Amylose:glycerol = 2.5:1.0. Film tested at 50% RH. Thickness of 0.07–0.10 mm. |
| Amylose | – | 10.0 | Lourdin, Della Valle, & Colonna, 1995. Amylose:glycerol = 5.0:1.0. Film tested at room conditions. |
| Wheat gluten | 29.1 | 4.1 | McHugh & Krochta, 1994. Gluten:glycerol = 5.7:1.0, film tested at 23 °C and 50% RH. |
| Wheat gluten | 13.9 | 30.8 | McHugh & Krochta, 1994. Gluten:glycerol = 2.3:1.0, films tested at 23 °C and 50% RH. |
| LDPE[d] | 7–16 | 100–800 | Bader & Göritz, 1994. |
| HDPE[d] | 26 | 300 | Salame (1986). Films tested at 23 °C and 50% RH. |

[a] Run 1 of the experimental design.
[b] Run 6 of the experimental design.
[c] Run 7, respectively, of the experimental design.
[d] LDPE—low-density polyethylene. HDPE—high-density polyethylene.

between thickness and glycerol concentration ($x_1x_2$) had a negative effect on strain at break (Table 2 and Fig. 4b), thus, the lower value for this response was obtained for the film formulated with the lower thickness and glycerol concentration (run 1 of the experimental design, Table 2); on other hand, this formulation originates the film with the higher tensile strength value (Fig. 3).

As seen in Tables 1 and 3, stress at break of yam starch films ranged from 2.84 to 7.84 MPa and strain at break ranged from 10.3 to 47.0%. The tensile stress values were slight lower, but comparable, to those of other hydrophilic and synthetic films while strain values were much lower than those of synthetic films and comparable to those of other hydrophilic films (Table 3).

Young's modulus or elastic modulus is the fundamental measure of the film stiffness, as higher Young's modulus, higher the stiffness of the material. According to ANOVA (Table 2), Young's modulus was affected by effect of thickness ($x_1$) and glycerol concentration ($x_2$) and by the three interaction factors ($x_1x_2$), ($x_1x_3$), ($x_2x_3$). Lower values for this response were obtained at higher thicknesses and glycerol concentrations, and the effect of glycerol was more pronounced in films with lower thicknesses (Fig. 5). This data was in agreement with the effect of glycerol as a plasticizer, improving the flexibility and extensibility of starch films.

The Young's modulus ranged from 201 to 28 MPa (Table 1), values comparable to that of low density polyethylene (LDPE) film, that shows values from 100 to
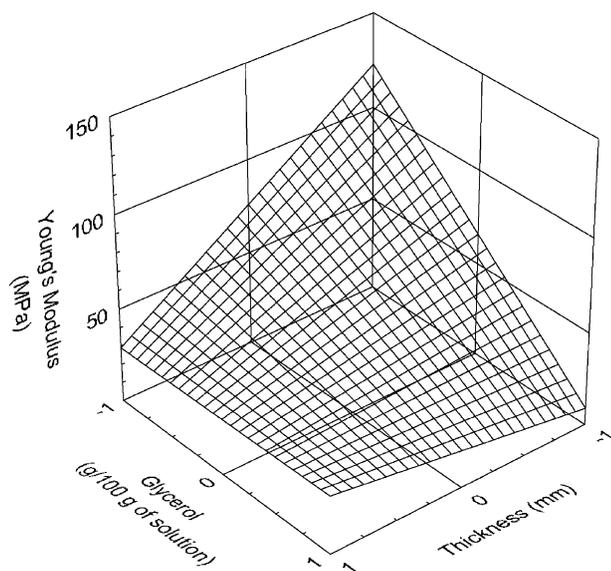


Fig. 5. Response surface for the effects of thickness and glycerol concentration, at a fixed glycerol concentration of 1.65%, on Young's Modulus of yam starch films.
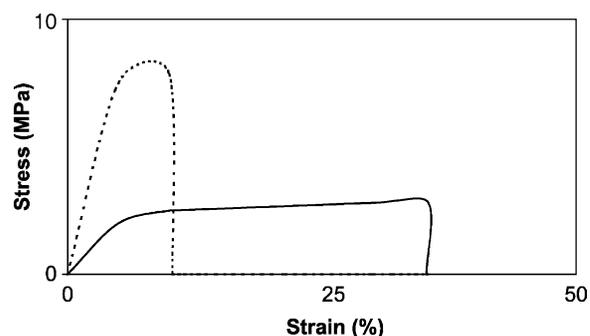


Fig. 6. Stress–strain curves of yam starch films: (---) film formulated with 1.3% of glycerol and 3.3% of starch and (—) film formulated with 2.0% of glycerol and 3.3% of starch.
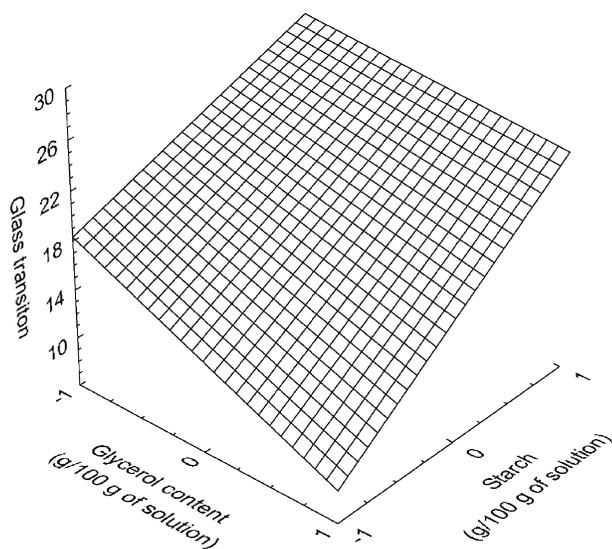
Fig. 7. Effect of glycerol and starch content on glass transition temperature ($T_g$) of yam starch films.

240 MPa (Doak, 1986); the film that showed the maximum value combined the lower thickness and glycerol concentration (run 1 of the experimental design, Table 1), the same formulation that presented the higher tensile stress and the lower strain at break. The film with the minimum value contained the highest glycerol/starch ratio (2.00/3.30—run 7 of the experimental design, Table 1). The influence of glycerol concentration on these films could be easily observed when looking at the primary tensile results (Fig. 6), the tensile stress–strain curves show the more brittle character of the film prepared with lower glycerol concentration (run 1 of the experimental design, Table 1) compared to the behavior of the film with the highest glycerol/starch ratio (run 7 of the experimental design, Table 1).

### 3.4. Thermal properties

In Fig. 7 are presented the glass transition temperature ($T_g$) of yam starch films. Films formulated with low yam starch and high glycerol concentrations showed lower $T_g$ values and $T_g$ of the film formulated without plasticizer (control sample) was higher than those of films with glycerol (44.2 °C); the plasticizer decreased $T_g$ because it facilitates chain mobility. The lowest $T_g$ (8.8 °C) corresponded to runs 3 and 7 of the experimental design (Table 1), with the highest glycerol/starch ratio, explaining the highest deformation values of this samples. Considering that our films are under rubbery state at ambient temperature, this behavior could be related to the structural modifications of starch network when glycerol was incorporated, the matrix of the film become less dense and under stress movements of polymer chains were facilitated. Plasticizers are expected to decrease intermolecular forces

between polymers chains and reduce $T_g$, imparting increased film flexibility while decreasing barrier properties (McHugh & Krochta, 1994).

Glass transition occurs below or above ambient temperature as a function of plasticizer and water content; water is a plasticizing agent able to decrease $T_g$ in high molecular weight biopolymers at contents around or higher than 30% dry basis (Slade & Levine, 1993; Lourdin, Coignard, Bizot, & Colonna, 1997). The water content of yam starch films (Table 1) ranged from 23.63 to 27.72 (g water/100 g dried film) and could be observed that glycerol and starch concentrations did not influence the water content, certainly because its variation was not sufficient to increase significantly the water content at 64% RH. These $T_g$ and water content data agreed with those of Chang et al. (2000), who worked with tapioca starch films; they stressed that at water content below 27% (w/w), glass transition of starch films would be expected to be below at 30 °C.

### 4. Conclusion

Yam starch films were studied with regard to their mechanical properties and the relationships of these properties with $T_g$ of the films. Film formulation (mainly glycerol concentration) and film thickness had a marked influence on mechanical properties and glycerol behaved as a typical plasticizer in starch films, with increasing concentration, puncture deformation and strain at break increased, and puncture force, tensile stress and Young's Modulus decreased. Depending on formulation, yam starch films can have tensile strength similar to low-density polyethylene films but deformation was always lower. Glass transition temperatures of films without glycerol were higher than those of films with plasticizer as measured by TMA, confirming its plasticizing effect.

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