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Mechanical properties of cassava starch films as affected by different plasticizers and different relative humidity conditions

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Abstract

The influence of plasticizer type (glycerol and sorbitol), its content (starch:plasticizer ratio of 1:0.15; 1:0.20; 1:0.25 and 1:0.30) and the relative humidity conditions (43, 58, 75 and 85%) on the mechanical properties of cassava starch films was studied. Both plasticizers seemed to integrate homogeneously in the film matrix. The incorporation of different concentrations of plasticizers affected the mechanical properties of the cassava starch films. Plasticizer ratio directly influenced the force values of the films, they showed significant flexibility when the plasticizer proportion was increased in the formulation. Under conditions of low relative humidity, sorbitol produced films more resistant to puncture than glycerol. Qualitatively, all the films were less brittle when the plasticizers were incorporated.

Keywords: Edible films; Mechanical essays; Puncture tests; Glycerol; Sorbitol; Models

1 Introduction

Many studies about the development and/or applications of edible and/or biodegradable materials from biopolymers, products and by-products of agro materials and renewable sources, have been studied recently. Specifically, there is great interest in edible films or coatings, which imply the use of materials and food grade additives in their formulations. The main component of these materials is a biopolymer, which must be able to form a continuous matrix. The coating is produced directly on the product to be covered, whereas the edible film is produced on a support, to be taken off later. Thus, this material, which is a thin and flexible film, can be used to manufacture food packaging.

According to Bonilla, Atares, Vargas, and Chiralt (2013), biodegradable polymers based on natural polysaccharides, particularly starch, can be produced at low cost and on a large scale and their applications in food packaging are promising due to their environmental appeal, flexibility, transparency, thermoplastic properties and low cost. Starches from different sources like cassava, potatoes, corn, wheat and rice, natural or modified, have been used in the preparation of coatings (García, Martino, & Zaritzky, 1998) and edible films (Stading, Rindlav-Westling, & Gatenholm, 2001; Forssell, Partanen, Buleon, Farhat, & Myllarinen, 2002; Mali & Grossmann, 2003; Ollett, Parker, & Smith, 1991; Lourdin, DellaValle, & Colonna, 1995).

In particular, cassava starch (*Manihot esculenta C.*) is an important source in some countries like Brazil, which is the largest cassava producing country, as well as in Thailand, Malaysia, In-

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donesia and some regions of Africa (Zhu, 2014; Versino & Alejandra Garcia, 2014). This starch is a cheap and abundant material, able to form a continuous polymer matrix (Bergo, Freitas Moraes, & do Amaral Sobral, 2012; Bergo, Sobral, & Prison, 2010; Bergo, Sobral, Guevara, & Vadala, 2010; Bergo et al., 2008), even without plasticizer (Vicentini, Dupuy, Leitzelman, Cereda, & Sobral, 2005). It has attracted particular interest because it is able to form edible films and coatings (Vicentini & Cereda, 1999; Vicentini, Castro, & Cereda, 1999; Henrique & Cereda, 1999; Oliveira & Cereda, 1999) and flexible, tasteless, odourless, colourless, transparent, nontoxic and biologically degradable films (Chiumarelli & Hubinger, 2014; Belibi et al., 2014; Chang, Cheah, & Seow, 2000).

However, cassava starch films produced without plasticizers exhibit poor mechanical strength and become fragile with low water content (Vicentini et al., 2005). An alternative to avoid such brittle characteristic would be the use of plasticizers, such as some low molecular polyols (Liu, Xie, Yu, Chen, & Li, 2009; Mali, Sakanaka, Yamashita, & Grossmann, 2005; Ramírez et al., 2011; Gontard, Guilbert, & Cuq, 1993; Cuq, Gontard, Cuq, & Guilbert, 1997). Usually, increasing the concentration of plasticizer leads to reduction of mechanical resistance and rigidity and increasing deformability of films, but the behavior depends on the type of plasticizer used (Sobral, Menegalli, Hubinger, & Roques, 2001; Gontard et al., 1993; Gennadios, Park, & Weller, 1993; Cuq, Gontard, Cuq, & Guilbert, 1997; Debeaufort & Voilley, 1997). The concentration of plasticizers generally ranges from 10 to 60g/100g dry matter, depending on the rigidity of the polymer (Lazaridou & Biliaderis, 2002), while moisture content of the film is dependent on the relative humidity of the environment in which it is stored (Chang et al., 2000; Gennadios et al., 1993).

In this context, the aim of this work was to analyze the effect of glycerol and/or sorbitol incorporation on the mechanical properties of cassava starch films at different relative humidities.

2 Materials and Methods

2.1 Preparation of the films

Cassava starch (*Manihot esculenta C.*) was supplied by Flor de Lótus (São Paulo, Brazil), previously characterized by Vicentini, Sobral, and Cereda (2002). The plasticizers studied were glycerol ($C_3H_8O_3$, MW = 92 g/gmol) and sorbitol ($C_6H_{14}O_6$, MW = 182 g/gmol) provided by Nuclear U.S.P, Brazil.

The film-forming solutions (FFS) were prepared with 2g starch/100g of water weighed in a semi-analytical balance (±0.01g) (Mars, AS2000). The FFS were heated at 70°C in a water bath (Tecnal, TE 184) for 40 min with constant mechanical stirring (Tecnal TE 039) to promote starch gelatinization. Plasticizer was afterwards added to the starch solution in starch:plasticizer ratios of 1:0.15; 1:0.20; 1:0.25 and 1:0.30. The FFSs (60 g) were cast in petri dishes polycarbonate (139.2 cm^2) and dried in an oven (Tecnal, MA037-3) with the temperature (30°C ± 0.5) and relative humidity (55-65%) controlled. These techniques have been used in previously works (Bergo, Sobral, & Prison, 2010; Bergo et al., 2008).

Prior to characterization, the films were conditioned for 6 days at 25°C in desiccators containing saturated solutions of K_2CO_3, NaBr, NaCl, KCl which provided environments with a relative humidity (RH) of 43, 58, 75 and 85%, respectively (Bergo et al., 2012).

2.2 Thickness of the films

The film thickness was measured using a digital micrometer (Mitutyo, Japan) to the nearest 0.001 mm. Nine random positions in each film samples were measured.

2.3 Moisture of the films

Once equilibrated, film samples were taken from each formulation to determine the moisture content by means of a gravimetric method. Firstly, 1g of film was weighed using an analytical balance (Scientech SA-210) to the nearest 0.0001g. The samples were dried in an oven at 105°C for

24h until constant weight, as described by (Bergo et al., 2012). The moisture content (MC) was expressed as the g of water/100 g of the dried film. The results from at least four samples were averaged.

2.4 Mechanical properties

Mechanical properties of the films were determined by puncture tests, using a Universal Testing Machine (TA.XT2i Stable Micro System) at room temperature (25°C). Four replicates were carried out for each formulation and test.
The puncture force (PF) and deformation (PD) were determined according to the methodology established in the literature (Cuq, Gontard, & Guilbert, 1997; Gontard & Ring, 1996). The films were fixed in cells with 52.6 mm diameter aperture and perforated by a probe of 3 mm diameter, moving at 1 mm/s. The puncture force (PF, N) and displacement were obtained directly from the force curves as a function of probe displacement. Then, puncture deformation (PD, %) was calculated from the distance travelled by the probe to the puncture displacement.

2.5 Statistical analysis

The statistical analysis of the data was performed by analysis of variance (ANOVA) using Statgraphics Plus (Manugistics Corp., Rockville, MD). Fisher's least significant difference (LSD) procedure was used to compare the means. Mechanical values were fitted to multiple regression equations using the design factors (concentration of each plasticizer and relative humidities) as independent variables, using the Statgraphics program.

3 Results and Discussion

3.1 Thickness of cassava starch films

The results of the thickness of the films conditioned at 25°C and four different relative humidities (43, 58, 75, 85%), with the incorporation of two different plasticizers (glycerol and sorbitol) are shown in Table 1.
Although all films were cast at a constant mass/surface ratio (60 g solids/cm^2), the final thickness of the films conditioned at the same relative humidity differed significantly ($p < 0.05$) as a function of the starch/plasticizer. The film thickness tended to increase when the starch ratio decreased in the film. The behavior regarding the plasticizer was explained by the fact that when the solution was more concentrated in plasticizer, a larger amount of dry matter (starch + plasticizer) was added, thereby resulting in increased thickness of the films, as described other authors (Sobral, 2000; Vicentini et al., 2002). The greater changes in the thickness in films with glycerol incorporated were presented in the films conditioned at 75% HR (from 0.064 ± 0.005 to 0.084 ± 0.006). In the case of the films with sorbitol added, the greatest changes in the thickness were at 58% HR (from 0.059 ± 0.005 to 0.081 ±0.005).
Moreover, the thickness of the films with glycerol incorporated at different RH did not differ significantly ($p > 0.05$). However, the films with sorbitol were significantly different ($p < 0.05$). These results were possibly due to sorbitol have more hydroxyl groups to interact with water by hydrogen bonds than glycerol.

3.2 Moisture of cassava starch films

The conditioning of the samples at different relative humidities caused water vapour adsorption of the films increasing its moisture content, as shown in Fig. 1. According to da Matta, Sarmento, Sarantopoulos, and Zocchi (2011), the plasticizer addition, in particular glycerol, has a great influence on starch films moisture, due to its hygroscopic character. Moreover, glycerol interacts with the film matrix by increasing the space between the chains, facilitates water migration into the film and, consequently, increases solubility.
In general in this study, the incorporation of glycerol made the films slightly more hygroscopic than when sorbitol was added. Molecular differences between glycerol and sorbitol were probably responsible for the different sorption be-

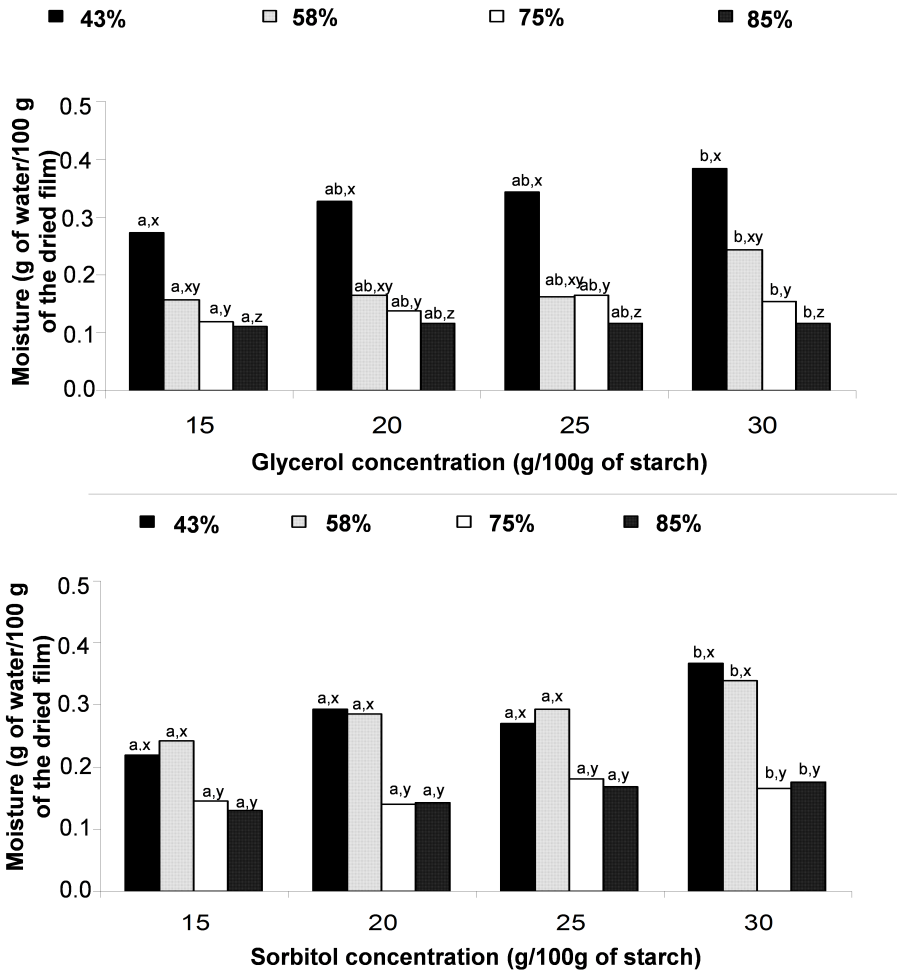

Figure 1: Moisture content of cassava starch films plasticized with glycerol and sorbitol. Values of concentration of plasticizer with the superscripts (a, b or, c) and relative humidity (x, y or x) were not significantly different in the LSD test

Table 1: Thickness[1] (mm) of cassava starch films with different plasticizers and conditioned in different relative humidities[2]

Plasticizer concentration	Relative humidity of conditioning			
	43%	58%	75%	85%
Glycerol				
15	0.069 (0.006) $^{a.x}$	0.070 (0.004) $^{a.x}$	0.064 (0.005) $^{a.x}$	0.064 (0.005) $^{a.x}$
20	0.067 (0.007) $^{ab.x}$	0.070 (0.004) $^{ab.x}$	0.082 (0.036) $^{ab.x}$	0.082 (0.036) $^{ab.x}$
25	0.070 (0.004) $^{ab.x}$	0.065 (0.005) $^{ab.x}$	0.074 (0.005) $^{ab.x}$	0.082 (0.005) $^{ab.x}$
30	0.071 (0.004) $^{b.x}$	0.086 (0.006) $^{b.x}$	0.084 (0.006) $^{b.x}$	0.079 (0.004) $^{b.x}$
Sorbitol				
15	0.073 (0.005) $^{a.xy}$	0.059 (0.005) $^{a.x}$	0.067 (0.004) $^{a.xy}$	0.081 (0.004) $^{a.y}$
20	0.079 (0.007) $^{ab.xy}$	0.070 (0.004) $^{ab.x}$	0.070 (0.004) $^{ab.xy}$	0.081 (0.004) $^{ab.y}$
25	0.073 (0.006) $^{ab.xy}$	0.074 (0.005) $^{ab.x}$	0.087 (0.006) $^{ab.xy}$	0.079 (0.004) $^{ab.y}$
30	0.076 (0.005) $^{b.xy}$	0.081 (0.005) $^{b.x}$	0.086 (0.007) $^{b.xy}$	0.085 (0.006) $^{b.y}$

[1] Mean values (standard deviation).
[2] Values of concentration of plasticizer (columns) with the same superscript (a, b or c) and relative humidity (rows) with the same superscript (x, y or z) were not significantly different in the LSD test

haviour of films plasticized by them. Glycerol and sorbitol are polyols with similar straight-chain molecules; however, the glycerol molecule is smaller (MW = 92 g/gmol) and has three hydroxyl groups, while the sorbitol molecule (MW = 182 g/gmol) has six hydroxyl groups. Although sorbitol presented more hydroxyl groups to interact with water by hydrogen bonds, glycerol presented higher water affinity demonstrated by adsorption and desorption isotherms. Since sorbitol is more similar to the molecular structure of glucose than glycerol, the chances of sorbitol interacting with polymeric starch chains are higher, thus, sorbitol containing films presented higher intermolecular forces and showed a lower capacity to interact with water (Mali et al., 2005). Similar results were reported by Mali et al. (2005) and Alves, Mali, Beleia, and Grossmann (2007), who also studied the influence of the plasticizer in cassava starch films.

3.3 Mechanical properties of cassava starch films

A food packaging generally requires high stress with deformation according to the intended application. In general, a food packaging must be an undeformable material to provide structural integrity or reinforce food structure, or even, a deformable film for other applications (Alves et al., 2007).

The results of puncture test are shown in Fig. 2. In general, it was observed that the films became more extendible when the concentration of plasticizer and relative humidity conditioning were increased. The reduction of the puncture force and the increasing of puncture deformation were consequences of the incorporation of plasticizers, and to water molecules absorbed by the samples, a common phenomenon of edible films, as has been revealed in other studies (Sobral et al., 2001; Cuq, Gontard, Cuq, & Guilbert, 1997; Debeaufort & Voilley, 1997; Ollett et al., 1991; Gennadios et al., 1993; Gontard et al., 1993). The significant increase in hydration of the films, produced by increase in relative humidity, caused a swelling of the biopolymer matrix, thus leading to an increase in the mobility of polymer chains. This made the bond strengths between them weaker, which consequently contributed to the decrease in strength and rigidity of the films (Chang et al., 2000; Cuq, Gontard, Cuq, & Guilbert, 1997; Gontard et al., 1993).

Comparing both plasticizers, it was observed that, in general, and especially at lower relative humidity, the films were more resistant to puncture obtained by using sorbitol, with the force

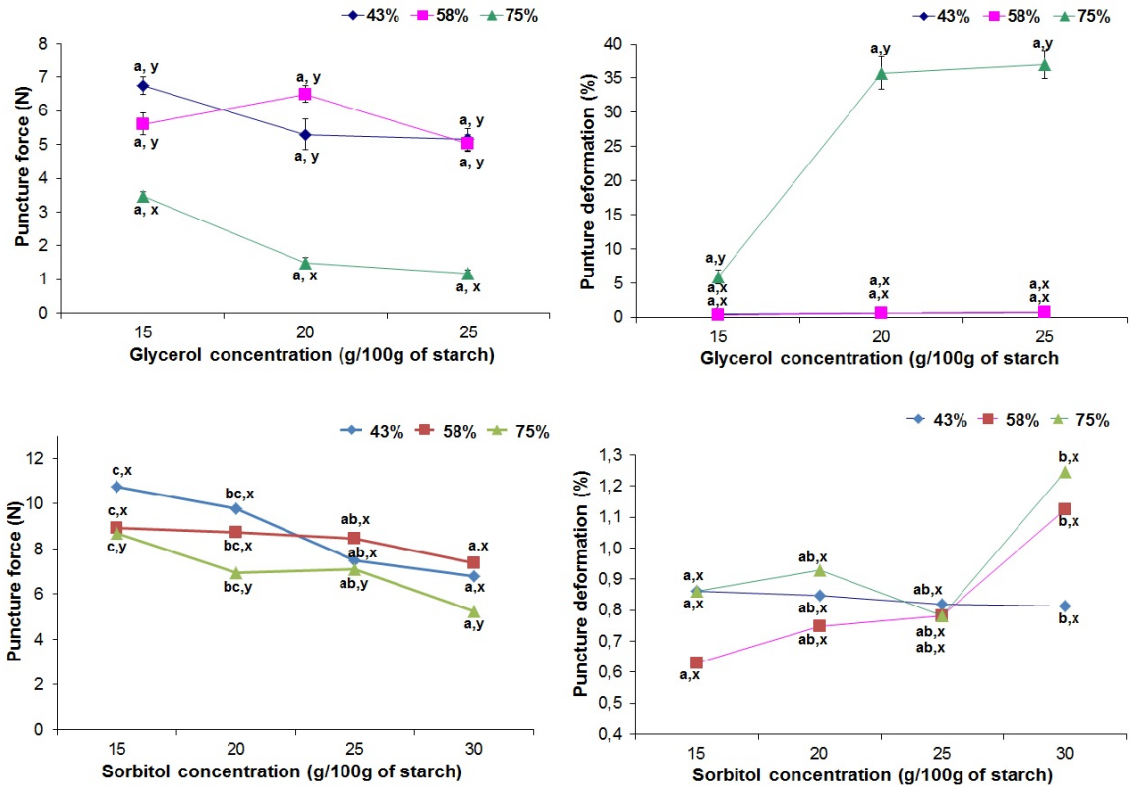

Figure 2: Mechanical properties of cassava starch films plasticized with glycerol and sorbitol. Values for concentration of plasticizer with the same superscript (a, b or c) and the relative humidity (x, y or z) were not significantly different in the LSD test

values at the puncture between 3 and 12 N, while the least resistant were those made with glycerol, with values between 1 and 8 N. This behaviour was consequence of the higher plasticizer effect of glycerol.

All films conditioned at lower relative humidity showed very small puncture deformation, practically independently of the concentration of plasticizer. In conditions of high relative humidity (75%), the films with glycerol showed higher puncture deformation values (variation between 15 and 30g plasticizer/100 g starch), resulting in more deformable films, with values ranging from 1 and 32%. The opposite results were obtained with sorbitol. This behaviour could be explained by the increase of the molecular weight from glycerol to sorbitol. Lourdin et al. (1995) observed that the addition of glycerol as plasticizer above 20% changes the mechanical behaviour of starch films by extending the plasticity zone.

Chen and Lai (2008) studied the puncture characteristics of tapioca starch/decolorized hsian-tsao (dHG) leaf gum films in the presence of glycerol. As glycerol concentration increased from 25% to 40%, the greater incorporation of glycerol into starch/dHG network resulted in reduction of puncture strength and an increase of puncture deformation. However, the formation of junction zones between starch and dHG still yielded films having higher puncture strength and lower puncture deformation than films without dHG.

According to Maran, Sivakumar, Sridhar, and Immanuel (2013), the puncture deformation was increased due to the increase in mobility of poly-

Table 2: Coefficients and significance of regression models to mechanical properties according to the concentration of plasticizers, moisture and thickness conditions for obtaining cassava starch films

Plasticizers	Term	Puncture force (PF, N)	Puncture deformation (PD, %)
Glycerol	Model	0.0109*	0.0160*
	Constant	0.0047*	0.0083*
	Concentration of plasticizers (g/100g of starch)	0.1046	0.2500
	Moisture (%)	0.0078*	0.0353*
	Thickness (mm)	0.1325	0.0277*
	R^2 (%)	92.2585	90.616
Sorbitol	Model	0.0024*	0.8366
	Constant	0.0006*	0.3256
	Concentration of plasticizers (g/100g of starch)	0.0014*	0.6931
	Moisture (%)	0.0030*	0.8201
	Thickness (mm)	0.0168*	0.4390
	R^2 (%)	96.4299	17.47

* Level of significance (95.0%)

mer chains in the presence of glycerol in the tapioca starch films. The permeated glycerol molecules could further decrease the interactions among the starch macromolecules, because of the formation of hydrogen bonds between the hydroxyl groups of starch macromolecules and glycerol small molecules. These all are favourable to the movement and rearrangement of the macromolecule chains of the starch, which caused the increase in the flexibility of the starch films. On other hand, puncture force was enhanced with increasing starch content. Sobral et al. (2001) observed that puncture deformation values of gelatin film increases with increasing plasticizer content. In that study, puncture force and deformation varied between 5.6–13.2 N and 3.5–4.7 mm respectively.

Table 2 shows the results of adjusting a multiple linear regression model to describe the relationship between mechanical properties (puncture force and puncture deformation) and three independent variables (concentration of plasticizers, thickness and moisture content). The equations of the fitted models applied on puncture force, using glycerol (Eq. 1) or sorbitol (Eq. 2) are presented. The R-square (R^2), 92.3% and 96.4% to the incorporation of glycerol and sorbitol respectively, indicated variability of the results with respect to this property.

$$PF = 19.9–0.13 \cdot Cg–101.2 \cdot X–0.10 \cdot M \quad (1)$$

$$PF = 12.1–0.33 \cdot Cs + 83.2 \cdot X–0.05 \cdot M \quad (2)$$

where Cg and Cs are concentrations (g/100g of starch) of glycerol and sorbitol, respectively; X is the thickness (mm) and M is the moisture content (%).

On other hand, only the equation of the model fitted to puncture deformation of data of films with glycerol was significant (5%) (Eq. 3). The R-square (R^2), 90.6% indicated the variability of the results with respect to this property.

$$PD = -151.1+0.73 \cdot Cg+1613.1 \cdot X+0.57 \cdot M \quad (3)$$

4 Conclusions

The incorporation of different concentrations of plasticizers affected the mechanical properties of the cassava starch films. Glycerol and sorbitol plasticizer addition made films more flexible and homogeneous. Under conditions of low relative humidity, sorbitol produced films more resistant to puncture than glycerol. In conditions of high relative humidity and higher concentration of

plasticizer, particularly glycerol, the films were more deformable at break and were slightly more hygroscopic. The increasing of relative humidity of conditioning and of the plasticizers concentration resulted in an increase of thickness of the films, principally in the case of the films with sorbitol incorporated.

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