MECHANICAL PROPERTIES, PERMEABILITY AND SOLUBILITY OF FILMS COMPOSED OF YAM STARCH AND GLYCEROL

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SUMMARY

The objective of this study was to evaluate the effect of yam starch (5-10%), plasticizer concentration (10-50%) and processing temperature (300-315K) on the mechanical properties, solubility and permeability of films. A rotational central composite design was used, utilizing the surface response method. The study concluded that the mechanical properties of yam starch and glycerol films were highly affected by the plasticizer content. Deformation and elongation properties were higher when utilizing lower temperatures and higher glycerol concentrations. As glycerol content decreased and drying temperature increased, the independent variable increased for rupture force. Tensile strength was affected by the interaction between starch and glycerol, with the plasticizer having the greatest effect. Low concentrations of plasticizer increased tensile strength in the films. Water solubility was affected by the interaction between starch and glycerol; higher levels of starch and glycerol increased solubility of the films. There was no significant interaction for permeability, while temperature and glycerol positively affected this property.

Introduction

The main function of packaging is to protect a product against mechanical damage and moisture loss (Chitarra and Chitarra, 2005) for the purpose of extending its shelf-life under storage. The incentive for researches in the development of biodegradable packages with features making them fit for use is due to the difficulty in recycling most available synthetic packages (Mali *et al*., 2010).

Continuously growing for more than 50 years, the global plastic production in 2012 rose to 288×10^{6}ton, a 2.8% increase from 2011. However, in Europe, in line with the general economic situation, plastic production decreased by 3% from 2011 to 2012. China remains the leading plastic producer with 23.9%, and the rest of Asia (including Japan) accounts for an additional 20.7%. European production accounts for 20.4% of the world's total production (Plastics Europe, 2013).

Due to negative impacts of synthetic plastic bags, like their short working life and low degradability (100 to 400 years to be degraded in the environment), visual pollution and obstruction of public drainages, beside its negative interference in wildlife feeding (Santos *et al*., 2012), there is great interest in developing edible or biologically degradable biofilms.

Biofilm characteristics are intimately related to the macromolecules used in their formulation and are dependent on the manufacturing process. In the casting technique, biofilms are obtained by drying a complex solution consisting of a polymer, a volatile solvent (and sometimes a non-volatile one) and, occasionally, a lipid phase (Tapia-Blácido *et al*., 2005).

In the manufacture of edible films and coverings, polysaccharides are the main raw material used. Starch, in particular, provides excellent sensory and mechanical qualities, and is biodegradable, it has a low production cost and is obtained from renewable resources (Thiré *et al*., 2004). Yam starch has a high amylose level, which makes it suitable for the extraction of this polymer (Silva *et al*., 2007).

According to Wittaya (2012), the native starch films are brittle compared with synthetic polymers such as polyethylene and, technically, they need to be plasticized. A plasticizer is a substance that is incorporated into rigid materials to increase their flexibility, workability, and dispensability.

In light of the above, the purpose of the present study was to evaluate the effect of starch and plasticizer concentration and processing temperature on mechanical properties in puncture and tensile tests, as well as to perform solubility and permeability tests of films prepared from yam starch and glycerol.

KEYWORDS / Biodegradability / Films Properties / Glycerol / Perforation / Tensile Strength / Yam Starch /

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PROPRIEDADES MECÁNICAS, PERMEABILIDAD Y SOLUBILIDAD DE LA PELÍCULA BIODEGRADABLE DE ALMIDÓN DE ÑAME Y GLICEROL

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RESUMEN

El objetivo de este trabajo fue estudiar el efecto de la concentración de almidón de ñame (5-10%), plastificante (10-50%) y temperatura de proceso (300-315K) sobre las propiedades mecánicas, solubilidad y permeabilidad de películas. Se utilizó el diseño rotacional central compuesto con el método de superficie de respuesta. De los resultados se concluye que las propiedades mecánicas de las películas de almidón de ñame y glicerol fueron altamente influenciados por el contenido de plastificante. La deformación y las propiedades de elongación obtuvieron valores más altos cuando se emplearon temperaturas más bajas y mayores concentraciones de glicerol. En cuanto a la resistencia a la rotura el efecto fue contrario, ya que al disminuir el glicerol y aumentar la temperatura de secado, aumentó la variable independiente. La tensión fue influenciada por la interacción entre el almidón y el glicerol, de manera que el factor que más influyó fue el plastificante; bajas concentraciones de éste aumentaron la resistencia al estiramiento de las películas. La solubilidad en agua fue influenciada por la interacción entre el almidón y el glicerol de manera que los niveles más altos de almidón y glicerol aumentaron la solubilidad de las películas. Para la permeabilidad ninguna interacción fue significativa, mientras que la temperatura y el glicerol influenciaron positivamente esta propiedad.

PROPRIEDADES MECÂNICAS, PERMEABILIDADE E SOLUBILIDADE DE FILMES BIODEGRADÁVEIS DE FÉCULA DE INHAME E GLICEROL

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RESUMO

O objetivo deste trabalho foi estudar o efeito da concentração de fécula de inhame (5-10%), de plastificante (10-50%) e da temperatura de processo (300-315K) sobre as propriedades mecânicas, de solubilidade e de permeabilidade dos filmes. Utilizou-se o delineamento rotacional central composto, como método de superfície resposta. Dos resultados conclui-se que as propriedades mecânicas dos filmes de fécula de inhame e glicerol foram altamente influenciados pelo teor de plastificante. Propriedades de deformação e elongação obtiveram maiores valores quando a interação entre menores temperaturas e maiores níveis de glicerol ocorreram. Já em relação a força na ruptura, o efeito foi contrário, a medida que o nível de glicerol decresceu e a temperatura de secagem aumentou a variável independente aumentou. A tensão foi influenciada pela interação entre a fécula e o glicerol, de forma que o fator que mais influenciou foi o plastificante, que em menores níveis aumentou a força de tensão nos filmes. A solubilidade em água foi influenciada pela interação entre fécula e glicerol de forma que maiores níveis de fécula e glicerol aumentam a solubilidade dos filmes. Para a permeabilidade nenhuma interação foi significativa, a temperatura e o glicerol influenciaram positivamente esta propriedade.

Materials and Methods

Raw Material

Yam starch was extracted from yam (cv. São Bento), according to Cereda *et al*. (2003), altering the concentrations of the reagents used (1% solution of ammonium oxalate, and oxalic acid at a ratio of 1:1 (w/w)). Glycerol was obtained from Merck (São Paulo, Brazil).

Starch characterization

Yam starch was characterized for moisture content, ash, lipids, protein, total fiber, total, reducing and non-reducing sugars and starch percentage (AOAC, 2000). All tests were performed in triplicate.

Production of edible films

Film forming solutions were prepared according to the formulations shown in Table I. The levels of the variables employed in each trial were determined from a rotational central composite design, with three replications at the central point, for a total of eleven trials, with five levels for each independent variable (yam starch content and glycerol content) for each temperature.

Solutions were then heated to 363K for 270s and, while

TABLE I
LEVELS OF YAM STARCH, GLYCEROL (CODIFIED AND REAL VALUES) AND MOISTURE CONTENT IN ORDER TO PREPARE FILMOGENIC SOLUTIONS FOR DRYING

Trial	Yam starch			Glycerol			Moisture (g)
	Codified value	(%)	Real value (g)	Codified value	(%)	Real value (g)	
1	-1.000	6.41	6.41	-1.000	15.86	1.02	92.57
2	-1.000	6.41	6.41	1.000	44.14	2.83	90.76
3	1.000	8.59	8.59	-1.000	15.86	1.36	90.05
4	1.000	8.59	8.59	1.000	44.14	3.79	87.62
5	-1.414	5.00	5.00	0.000	30.00	1.50	93.50
6	1.414	10.00	10.00	0.000	30.00	3.00	87.00
7	0.000	7.50	7.50	-1.414	10.00	0.75	91.75
8	0.000	7.50	7.50	1.414	50.00	3.75	88.75
9	0.000	7.50	7.50	0.000	30.00	2.25	90.25
10	0.000	7.50	7.50	0.000	30.00	2.25	90.25
11	0.000	7.50	7.50	0.000	30.00	2.25	90.25

TABLE II
MEAN AND STANDARD ERROR VALUES OF THE CHEMICAL CHARACTERISTICS OF YAM STARCH (CV. SÃO BENTO), DRY BASIS

Parameters	Values (%)
Moisture content	7.61 ±0.17
Ashes	0.92 ±0.00
Lipids	0.64 ±0.02
Proteins	3.21 ±0.11
Crude fiber	0.17 ±0.03
Total sugars	1.63 ±0.03
Reducing sugars	0.28 ±0.01
Non-reducing sugars	1.35 ±0.01
Starch	84.19 ±0.02

still hot, aliquots of 1ml were transferred to acrylic plates with an internal diameter of 8.8cm.

Drying was carried out in a forced air circulation oven (Marconi® MA 035). All treatments were subjected to temperatures of 300, 305, 310, and 315K. Subsequently, the plates were stored in desiccators containing silica gel at 394 ±27K for 24h.

The thickness of the produced biofilms was measured using a manual micrometer Tesa Isomaster® (±0.01mm), model Swisscom, with precision of 10^{-5}m.

Mechanical tests were conducted using a TA.HD texturometer using the 'Texture Expert Exceed' program, vers. 2.64 (Stable Micro System). Five replicates were analyzed for each trial. The parameters used for each test were chosen according to the ASTM D882-95 (1995).

The methodology proposed by Gontard *et al*. (1994) was used to perform the puncture test. Deformation at rupture was determined using Eq. 1.

$$D = \frac{(d^2 + l_0)^{\frac{1}{2}} - l_0}{l_0} \quad (1)$$

where D: deformation at rupture (%), d: distance penetrated by the probe (m), l_0: radius of the film surface (m).

Tensile strength was determined through force and deformation at rupture. For testing, samples were cut to 10cm length and 250mm width. The samples were then subjected to a tensile speed of $1{\times}10^{-3}$m·s^{-1}, starting from an initial probe distance of 8cm until the films were ruptured. Tension and elongation at rupture were obtained directly from the tensile curve as a function of elongation, knowing the initial dimensions of the samples.

Permeability to water vapor was determined gravimetrically according to the methodology proposed by Gontard *et al*. (1992). The analysis of water solubility was performed in accordance with the method proposed by Gontard *et al*. (1994).

In order to evaluate the effect of yam starch and glycerol content and temperature on the studied variables, response surface graphs were made by means of the model that obtained a non-significant fit. The lack of fit may not be significant because the tested model should fit the experimental data analyzed.

Results and Discussion

Starch characterization

Characterization of yam starch was carried out to verify the effect of its composition on mechanical properties, permeability and solubility of the film produced from this polysaccharide. These results are shown in Table II.

Since this compound is a starch, the quantities of the components encountered indicate that it was not altered by purification, thus favoring its appropriateness for formation of biodegradable films, due to the presence of proteins and lipids. Proteins are able to form cohesive and continuous matrices (Carvalho and Grosso, 2006).

Films of chemically modified proteins have been studied by several authors, and the type and amount of protein and reagent used may directly affect their mechanical and barrier properties (Charulatha and Rajaram, 2003; Carvalho and Grosso, 2006; Cortez-Vega *et al*., 2013). Therefore, reagents used for extraction of yam starch may contribute to the formation of films with better properties.

The amount of ash present in starch may be associated with the cultivars used, as well as yam residue impurities that were not previously separated during the starch extraction process. The protein content observed in this study was lower than that found by Liporacci *et al*. (2005). These authors studied extraction with the addition of oxalic acid and ammonium oxalate (1:1) at 10% in yam starch, obtaining 3.99 and 3.73% protein for two batches. According to Nunes *et al*. (2010), yam starch has more protein than starch from other plants.

Total crude fiber content found in this study for yam starch was greater than that encountered by Manzano (2007), who obtained 0.02% of total fiber for yam starch.

Almeida *et al*. (2013) assessed the lipid content of yam starch, clone Macaquinhos, from the region of Dourados, Brazil, and found 1.25% higher content than in this study. But according to the same authors, lipid is an interfering agent, since a lipid-amylose complex is formed and compromises some properties, in favor of the lipid content found in this study.

Edible films

In order to verify the effects of yam starch, glycerol content and temperature on the mechanical properties of yam starch and glycerol films, analysis of variance of the tests was carried out (Table III). In order to verify the behavior of each mechanical variable studied, response surface graphs were plotted and are shown in Figure 1.

TABLE III
VARIANCE ANALYSIS FOR DEFORMATION (D), RUPTURE FORCE (RF), TENSILE STRENGTH (TS) AND ELONGATION (E) DATA FOR FILMS OF YAM STARCH AND GLYCEROL

Variation sources	D (%)		RF (N)		TS (MPa)		E (%)	
	DF	MS	DF	MS	DF	MS	DF	MS
Temperature (T)	1	3863.51*	1	71.1891 *	1	1.74 *	1	2062.23 *
T²	-	-	-	-	-	-	-	-
Starch (S)	-	-	1	5.5969 *	-	-	1	17.07 *
S²	-	-	-	-	-	-	-	-
Glycerol (G)	1	2699.31*	1	200.2088 *	1	8.39 *	1	19843.60 *
G²	1	34.29 *	-	-	1	0.49 *	1	29.84 *
T × G	1	26.27 *	1	3.0409 *	-	-	1	21.09 *
T × S	-	-	-	-	-	-	-	-
S × G	1	48.85 *	-	-	1	0.02 *	-	-
Lack of adjustment	39	3.878 ns	40	0.3841 ns	40	0.008 ns	39	7.27 ns
Pure error	10	2.073	10	0.1625	10	0.003	10	2.82

*: F-test significant at 5% probability, ns: F-test non-significant at 5% probability, DF: degrees of freedom, MS: mean square.

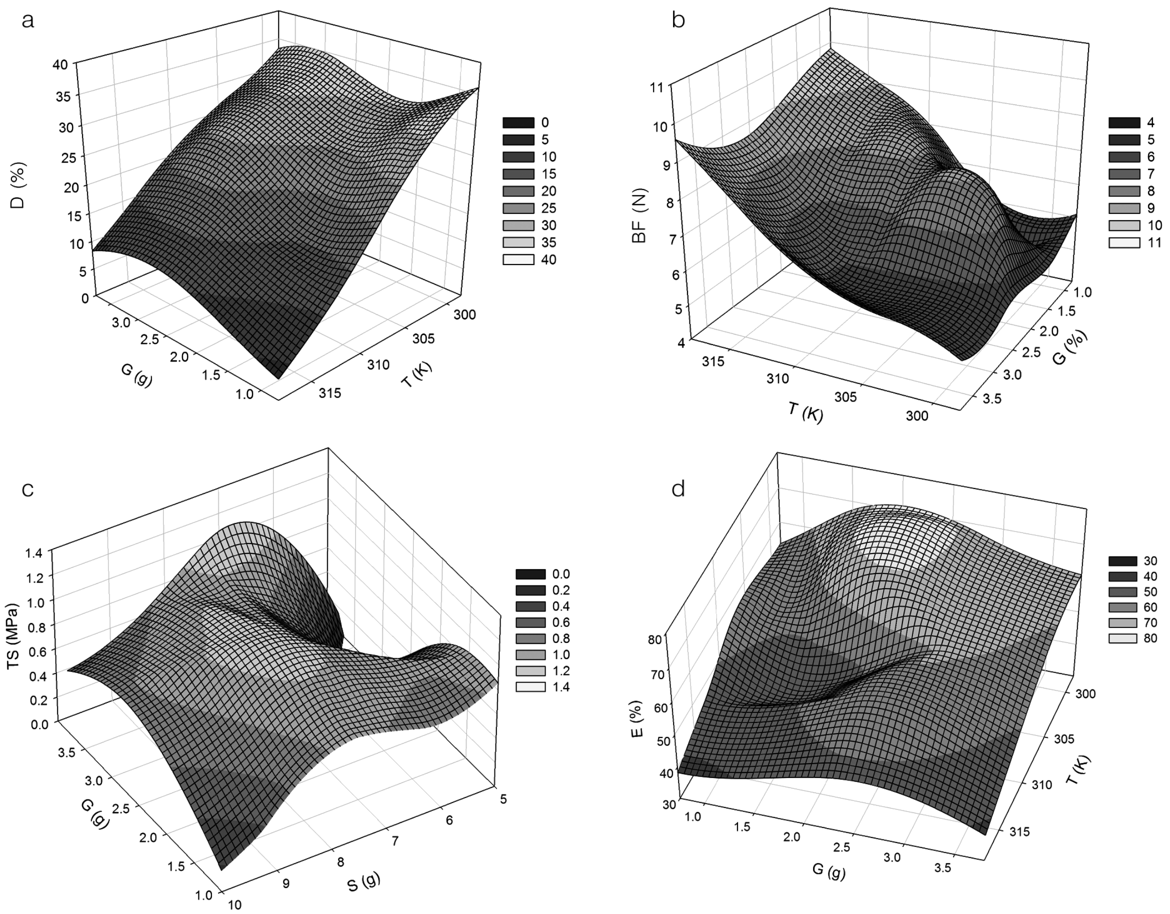

Figure 1. Surface deformation of the film (%) as a function of temperature (T) and glycerol content (G), fixing the starch content at 7.5%; b: breaking force (BF) as a function of temperature and glycerol content; c: tensile (TS) strength in function of starch and glycerol content; d: elongation (E) of the films as a function of temperature and glycerol.

Regression models were significant at 5% ($p<0.05$) and were expressed in the form of equations. They are shown in Eqs. 2 to 5, which represent the models for deformation, rupture force, tensile strength and elongation, respectively.

$$D= 43.95-0.96T+4.97G+1.04G^2-0.10TG \quad (R^2= 0.9678) \quad (2)$$

$$RF= 8.42+0.08T-3.09G+0.03TG \quad (R^2= 0.9140) \quad (3)$$

$$TS= 0.76+0.50T-1.22G+0.49G^2-0.15SG \quad (R^2= 0.9634) \quad (4)$$

$$E= 31.04-0.66T-0.48S+27.75G-0.82G^2-0.09TG \quad (R^2= 0.9863) \quad (5)$$

where G: glycerol content (g), T: temperature (K), and S: yam starch content(g).

Deformation in the yam starch and glycerol films was affected by the interaction between glycerol concentration present in the film forming solutions and drying temperature. Smaller amounts of glycerol together with higher temperatures produced films with less deformation.

Rupture force (RF) of the films was affected by the interaction between temperature and glycerol. It was observed that at higher concentrations of glycerol and lower temperatures, RF decreased. This tendency may be explained by the fact that higher levels of glycerol and lower temperatures make the biofilms more elastic, thus decreasing their RF and increasing deformation. According to Mali *et al.* (2010), mechanical properties of films are highly dependent on their formulation: macromolecules, solvent, plasticizer and pH adjustment.

The plasticizer reduces the molecular interactions between adjacent biopolymer chains used to manufacture the film, resulting in increased mobility of the chains and, consequently, providing more flexible materials. Thus, at the macroscopic level, alterations of all the physical or functional properties of the films can be observed (Gontard *et al.*, 1993; Sobral *et al.*, 2002). Starch films without plasticizers are resistant and elastic. Upon increasing the plasticizer content, these materials become more flexible and deformable (Mali *et al.*, 2004, 2005).

Bourtoom (2008) determined mechanical properties of the films made from rice starch and chitosanin with different plasticizers, and confirmed that the higher concentration of the plasticizers resulted in tension reduction and increase of the film elongation.

Tapia-Blácido *et al.* (2005) found a range of RF values between 0.73 and 1.60N, and 17.71 to 36.04% for deformation, in films composed of amaranth flour (4%) and glycerol (30-40%), while also varying the relative humidity of the stored product (40-70%). Lower RF values encountered by the above authors may be due to the high relative humidity of storage as compared to the films of the present study, which were stored in a desiccator. In regard to deformation, yam starch and glycerol films had a wider range, due to the greater variation in the amount of glycerol used.

Tensile strength (TS) at rupture of the products retrieved from drying of film forming solutions composed of yam starch and glycerol decreased with the increase of glycerol content in the solutions, obtaining values between 0.21 and 1.78MPa. This behavior was observed by Sobral (2000) when studying the effect of glycerol on the mechanical properties of films made from starch.

Shimazu *et al.* (2007) studied the effect of glycerol content on mechanical properties of cassava starch and glycerol films, with 3% and 0 to 40%, respectively, and water activity from 0.11 to 0.90. They observed values of tensile strength between 0.5 and 25MPa and also observed that films plasticized with glycerol tended to show a decrease in tensile strength as greater plasticizer concentrations were used. Tensile strength found by the above authors presented a wide range. Higher values were obtained for low water activity.

TABLE IV
VARIANCE ANALYSIS FOR THE SOLUBILITY (SOL) AND PERMEABILITY (P) DATA FOR FILMS OF YAM STARCH AND GLYCEROL

Variation sources	Sol (%)		P (g (m·h·Pa)$^{-1}$)	
	DF	MS	DF	MS
Temperature (T)	1	295.59 *	1	$8.77\times10^{-13*}$
T²	-	-	1	$2.45\times10^{-13*}$
Starch (S)	1	26.60 *	-	-
S²	1	6.24 *	-	-
Glycerol (G)	1	720.02 *	1	$2.84\times10^{-13*}$
G²	1	30.16 *	-	-
T × G	-	-	-	-
T × S	-	-	-	-
S × G	1	57.60 *	-	-
Lack of adjustment	38	3.11 ns	42	6.76×10^{-14} ns
Pure error	10	1.25	10	5.23×10^{-14}

*: F-test significant at 5% probability, ns: F-test non-significant at 5% probability.

Torres *et al*. (2011) studied mechanical properties of various films made from starches of different origins with 40% glycerol and confirmed that films from cassava and sweet potato starch had the highest elongation values at break, 43% and 33%, respectively. They also observed that the elongation at break values of starch films increases as their tension values decrease.

Finally, elongation of the biofilms in this work was affected by the interaction between temperature and glycerol. Higher concentrations of glycerol together with lower temperatures increased the elongation of the films, making them more flexible.

An analysis of the mechanical properties of films made from starch with high and low amylose content confirmed that the film tension is directly proportional, and the film elongation inversely proportional, to the amylose content (Muscat *et al*., 2012).

Analysis of variance of the response surfaces for solubility in water and permeability in water vapor of the films as a function of the independent variables studied are shown in Table IV and Figure 2, respectively.

Solubility of the films analyzed in this study was affected by the interaction between starch and glycerol, where the model that best fit observed data was one of second order plus interaction. As for permeability, the best fit model was a linear one with no interaction, where temperature and glycerol affected permeability separately. The models representing the behavior of the solubility and permeability variables are shown in Eqs. 6 and 7.

$$Sol= 16.60+0.33T+0.23S^2-12.75G+0.94G^2+1.67FG \quad (R^2= 0.9170) \quad (6)$$

$$P= 2.15\times10^{-7}-1.44\times10^{-7}T+2.28\times10^{-9}T^2+8.17G \quad (R^2= 0.4229) \quad (7)$$

Water solubility of yam starch and glycerol films was affected by the interaction between starch and glycerol. Greater solubility was found for higher levels of glycerol and lower yam starch. Lower levels of this variable were found for lower concentrations of glycerol and higher starch contents. It was observed that glycerol concentration had a major effect on water solubility of the films, since up to 2g of total glycerol, the region corresponding to variations in starch has lower solubility values. At glycerol concentrations >3g and starch concentrations of 7g, higher values can be observed.

Prates and Ascheri (2010) examined films of *Solanum lycocarpum* starch and sorbitol (0.1-0.3%) and found values of solubility in water between 26 and 29%, with a strong effect of the plasticizer content, which when increased causes increased solubility due to its hydrophilic characteristic.

The fact that a lower solubility was encountered in the present study may be related to the fact that the yam starch used for preparation of the films is not easily soluble in water. This property indicates the applicability of the biofilm as packaging for food products. In some cases, its complete solubility in water can be beneficial, as in prepackaged products ready for cooking. However, when the food is in a liquid state or exudes an aqueous solution, highly soluble biofilms are not recommended (Fakhouri *et al*., 2007). Thus, depending on the solubility characteristics of the films studied, they may be applied to fresh vegetables.

Solubility affects the ability of the biodegradable film to act as a barrier to water vapor. To obtain a low permeability to water vapor (within a wide range of relative humidity), it is necessary to use insoluble materials or materials that are not easily soluble in water (Fakhouri *et al*., 2003).

According to Sobral (2000), highly permeable material s, as is the case of starch films, may be suitable for packaging of fresh vegetables, while low permeability films are more suitable for dehydrated products. Permeability in this study ranged from 1.02×10^{-7} to 9.87×10^{-7}g·m^{-1}·h^{-1}·Pa^{-1}. There was no significant interaction for permeability; however, each factor contributed individually. Upon increasing the quantity of glycerol, permeability also increased, as was expected due to its affinity to water. The same behavior was observed due to the effect of temperature.

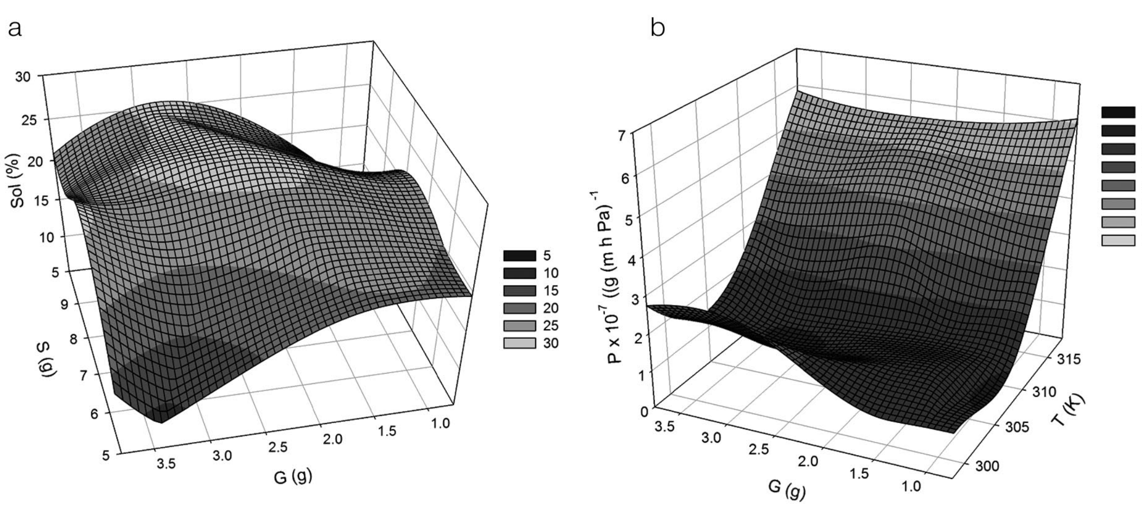

Figure 2. a: Surface solubility in water (Sol) of the yam starch and glycerol film, fixing the temperature at 35°C; b: water permeability (P) as a function of temperature and glycerol for a fixed yam starch content of 7.5%.

Ferreira *et al.* (2009), when analyzing biodegradable films produced by extrusion of yam starch and glycerol, found a mean value of 1.7×10^{-10}g·m^{-1}·s^{-1}·Pa^{-1}; in other words, 6.12×10^{-7}g·m^{-1}·h^{-1}·Pa^{-1} for films composed of film forming solution with 4g of starch and 1.3g of glycerol. This value is similar to that obtained in the present study.

The increase in temperature also resulted in increased permeability. According to Donhowe and Fennema (1994), an increase in temperature leads to an increase in permeability to water vapor, and these variations are dependent on the moisture content of the material.

A film with good barrier properties may be inefficient if its mechanical properties do not allow for maintenance of the integrity of the film during the processes of handling, packaging and transportation. Thus, edible films for packaging should have adequate resistance to breaking and abrasion, so as to provide good protection to food without losing quality through handling (Tanada-Palmu *et al*., 2003), and they should also be flexible, so as to adapt to deformations of the food without mechanical damage.

Conclusions

The mechanical properties of films composed of yam starch and glycerol were highly affected by the plasticizer. Films prepared with intermediate values of both glycerol and temperature meet the rupture force and tensile strength requirements, as well as deformation and elongation requirements. As for permeability to water vapor, intermediate values of temperature and glycerol are also suitable for use with fresh vegetables.

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