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Water sorption and mechanical properties of cassava starch films and their relation to plasticizing effect

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Abstract

Effects of plasticizers (glycerol, sorbitol, and 1:1 mixture of glycerol and sorbitol) on moisture sorption characteristics of cassava starch films were investigated at three levels of plasticizer concentration (0, 20, and 40 g/100 g starch). The combined effects of relative humidity and plasticizer on mechanical properties of starch films were also examined. Water affinities of cassava starch films were affected by hydrophilicity of the plasticizer and its concentration. Films plasticized with glycerol, under all RH conditions, adsorbed more moisture with higher initial adsorption rate, and films with higher plasticizers contents exhibited higher equilibrium moisture contents. Mechanical properties were affected by plasticizing effect, including the water adsorbed, resulting in higher strain and Young's modulus values for starch films and, in all cases, glycerol exerted a more effective plasticization.

Keywords: Biodegradable films; Glycerol and sorbitol

1. Introduction

Biopolymer films and coatings from polysaccharides, proteins and lipids, formulated either with one or more components, have potential to control mass transfer and thus extend food shelf life (García, Martino, & Zaritzky 1999, 2000; Parris, Coffin, Joubran, & Pessen, 1995). As being totally degradable, biopolymers could contribute to reducing the amount of plastic wastes. In addition, these polymers are obtained from renewable sources unlike the 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 works dealt 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; Mali, Grossmann, García, Martino, & Zaritzky, 2002, 2004; Souza & Andrade, 2000).

Plasticizers increase film flexibility due to their ability to reduce internal hydrogen bonding between polymer chains while increasing molecular space. The most effective plasticizers will generally resemble most closely the structure the polymer they plasticize, thus, the most commonly plasticizers used in starch-based films are polyols, such as sorbitol and glycerol. They avoid cracking of the film during handling and storage (Gontard, Guilbert, & Cuq, 1993), affect gas, water vapor and solute permeabilities (Banker, 1966) and sorption characteristics.

The moisture content in starch films can also affect significantly the physical and barrier properties of starch-based films; due to their inherent hydrophilic nature, starch films tend to absorb large quantities of water at elevated relative humidity (RH) conditions. Although there are studies focused on sorption properties of edible films, little information is available on the effect of type of plasticizer on sorption characteristics of these films (Cho & Rhee, 2002).

Water sorption isotherm equations are useful for predicting water sorption properties of hydrophilic films; they provide little insight into the interaction of water

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and film components. There are several mathematical models to describe water sorption isotherms of food systems materials, but no one gives accurate results throughout the whole range of water activities, or for all types of foods systems (Al-Muhtaseb, McMinn, & Magee, 2004). Labuza (1980) attributed this to the fact that the water is associated with the food matrix by different mechanisms in different water activity regions. Models available in the literature to describe moisture sorption isotherm can be divided into several categories; kinetic models based on a monolayer (BET model), kinetic models based on a multilayer and condensed film (GAB model), semi-empirical (Ferro-Fontan, Henderson and Halsey models) and empirical models (Smith and Oswin models) (Al-Muhtaseb et al., 2004). In more recent years, the Guggenheim, Anderson and de Boer (GAB) isotherm equation has been widely used to describe the water sorption behavior of foods (Bizot, 1984) and according to Al-Muhtaseb et al. (2004), GAB parameters are more representative in explaining the hydration process in foods and foodstuffs.

The objectives of this study were to evaluate the effect of plasticizer type and concentration on kinetic and static moisture sorption characteristics and on mechanical properties of cassava starch films.

2. Materials and methods

2.1. Materials

Cassava starch (19% amylose) was provided by Hiraki Industry (São Paulo, SP, Brazil). Glycerol and sorbitol were purchased from Synth (Diadema, São Paulo).

2.2. Films preparation

Cassava starch films were prepared by casting employing 3 g of starch/100 g of filmogenic solution; three types of plasticizer (glycerol, sorbitol, and 1:1 mixture of glycerol and sorbitol) were added at three levels (0, 20 and 40 g/100 g of starch). For each experiment, the quantity of filmogenic solution poured onto the acrylic plates was calculated to obtain a constant weight of dry matter of approximately 9 mg/cm^2, resulting films with 0.08 ± 0.01 mm of thickness, measured with a Mitutoyo micrometer (São Paulo, Brazil). The plates with the filmogenic solution were dried at 40 °C in a ventilated oven model TE-394-3 (Tecnal, Brazil) to constant weight (about 20 h). The result was translucent films, which could be easily removed from the plate.

2.3. Moisture sorption characteristics

2.3.1. Moisture curve and rates

Cassava starch film specimens (30 mm × 30 mm) were pre-dried for 7 days over phosphorous pentoxide (P_2O_7) and then were placed at 25 ± 2 °C over saturated salt solutions in separated desiccators having desired relative humidity (32, 58 and 90% RH) conditions (Rockland, 1960). Weights of film specimens were measured as a function of time, and moisture content of the films were determined by oven drying at 105 °C. Moisture adsorption data were fitted according to a mathematical model suggested by Peleg (1988): $M_{(t)} = M_0 + (t/(k_1 + k_2 t))$, where $M_{(t)}$ is the moisture after time, M_0 is the initial moisture content and k_1 is the Peleg rate constant (h/(g water/g solids)) and k_2 is the Peleg capacity constant (g solids/g water). All tests were conducted in triplicate.

2.3.2. Sorption isotherms

Cassava starch film specimens (30 mm × 30 mm) were pre-dried for 7 days over phosphorous pentoxide (P_2O_7) and then were placed at 25 ± 2 °C over saturated salt solutions in separated desiccators having desired relative humidity (11, 32, 58, 75 and 90% RH) conditions (Rockland, 1960). Each film specimen was weighted at regular intervals (minimum 1 h and maximum 6 h), and when two consecutive weightings were equal, it was assumed that an equilibrium condition was reached. Under the above conditions, an equilibrium period of 3 days was sufficient to establish moisture equilibrium. Equilibrium moisture content was calculated from the increase in mass of the dried sample after equilibration at a given RH. All tests were conducted in triplicate.

GAB (Guggenheim–Anderson–de Boer) model was used to fit cassava starch film sorption isotherm data, and monolayer values were calculated from the equations (Bizot, 1984). GAB isotherm model can be expressed as follows: $M = m_0 C K a_w/(1 - K a_w)(1 - K a_w + C K a_w)$, where M is the equilibrium moisture content at a water activity (a_w), m_0 is the monolayer value (g water/g solids) and C and K are the GAB constants.

2.4. Mechanical properties of starch films

Five film specimens (100 mm × 25 mm) of each formulation were pre-dried for 7 days over phosphorous pentoxide (P_2O_7) and then were placed at 25 ± 2 °C over saturated salt solutions in separated desiccators having desired relative humidity (32, 58, 75 and 90% RH) conditions (Rockland, 1960). The tensile properties were determined using a TA.TX2i Stable Micro Systems texture analyzer (Surrey, England) in accordance with ASTM D-882-91 method (1996). The samples were clamped between pneumatic grips and force (N) and deformation (mm) were recorded during extension at 50 mm min^{-1} and with an initial distance between the grips of 50 mm. The parameters determined were: stress at break (MPa) and Young's Modulus (MPa).

2.5. Statistical analysis

Analysis of Variance (ANOVA), Tukey mean comparison test ($p \leq 0.05$) and non-linear regression procedure were

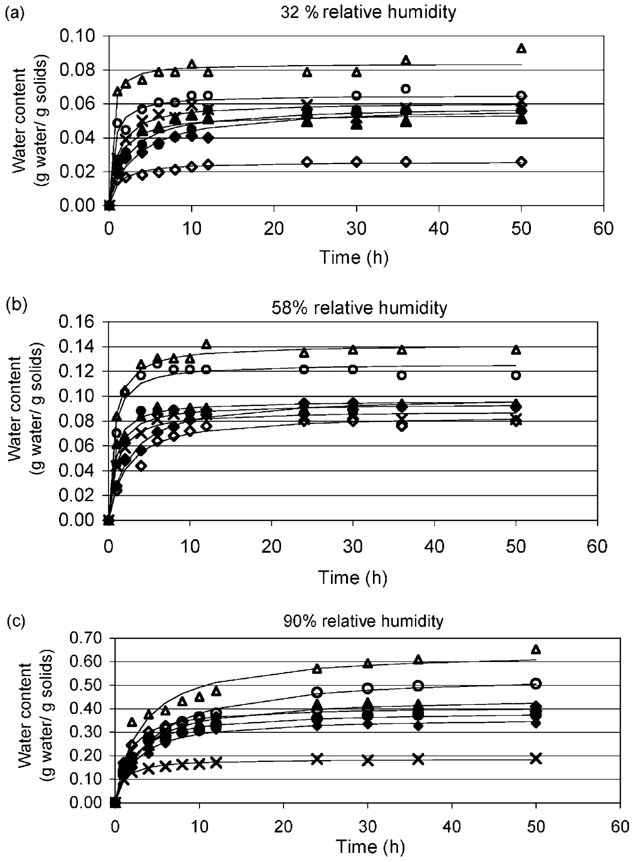

Fig. 1. Effect of plasticizers on moisture sorption curves of starch films at various RH. △, 40 g glycerol/100 g starch; ▲, 20 g glycerol/100 g starch; ○, 40 g glycerol:sorbitol/100 g starch; •, 20 g glycerol:sorbitol/100 g starch; ◇, 40 g sorbitol/100 g starch; ◆, 20 g sorbitol/100 g starch and ×, films without plasticizer. Lines are derived from Peleg's Model (Peleg, 1988). Coefficient of variation of analysis was 10%.

performed employing Statistica software (Statsoft, OK, USA).

3. Results and discussion

3.1. Moisture adsorption kinetics of cassava starch films

Moisture adsorption curves of cassava starch films are shown in Fig. 1. Moisture adsorption was more rapid at the initial stages of the storage and lower amounts of water were adsorbed as time increased. Then, the moisture content of cassava starch films reached a plateau indicating that they became equilibrated with storage RH. Moisture equilibrium time was influenced by storage RH and plasticizer type and concentration; films stored at 90% needed more time to reach the equilibrium and films plasticized with glycerol showed a trend to adsorb more water than sorbitol films, equilibrating faster too (Fig. 1). To improve the investigation about water sorption behavior, the moisture content data obtained at different times were fitted using Peleg model (Peleg, 1988).

The Peleg parameters (Peleg, 1988), k_1 and k_2, are shown in Table 1. As k_1 is a constant related to mass transfer, the lower k_1, the higher the initial water adsorption rate; k_2 is a constant related to maximum water adsorption capacity and the lower the k_2, the higher the adsorption capacity (Turhan, Sayar, & Gunasekaran, 2002). Films plasticized with the highest level of glycerol and stored at highest RH had lower k_1 and k_2 values (Table 1), indicating that these films adsorbed faster and more water during its storage. Films with mixtures of glycerol:sorbitol showed intermediate k_1 and k_2 values generally nearly that of films containing only glycerol (Table 1). According to Sothornvit and Krochta (2001), molecular differences between glycerol and sorbitol are probably responsible for the different sorption rate of films plasticized with them. Glycerol and sorbitol are polyols with similar straight-chain molecules; however, glycerol molecule is smaller (molecular weight of 92) and has three hydroxyl groups while sorbitol molecule (molecular weight of 182) 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 (Leung, 1986). According to García et al. (2000), since sorbitol is more similar to the molecular structure of glucose units than glycerol, the chances of sorbitol to interact with polymeric starch chains are higher, thus, sorbitol-containing films presented higher intermolecular forces and showed a lower capacity to interact with water.

Cho and Rhee (2002), working with soy protein films, observed the same trend for k_1 and k_2 Peleg parameters of sorbitol and glycerol plasticized films. Films with 50 g

Table 1
Constant values (k_1 and k_2) and coefficient of determination (r^2) for sorption curve equations (Peleg model)[a] of cassava starch films at selected RH

Plasticizer	Plasticizer level (g/100 g starch)	32% RH			58% RH			90% RH		
		k_1	k_2	r^2	k_1	k_2	r^2	k_1	k_2	r^2
Without	0	17.09	16.34	0.98	10.05	11.28	0.98	5.29	5.38	0.99
Glycerol	20	19.72	18.33	0.90	6.98	10.11	0.99	6.34	2.31	0.99
	40	3.48	11.98	0.98	4.59	7.04	0.99	5.37	1.61	0.96
Glycerol:sorbitol	20	42.73	18.07	0.94	8.51	10.53	0.98	5.46	2.59	0.99
	40	7.77	15.42	0.96	4.40	7.90	0.97	4.64	1.86	0.99
Sorbitol	20	64.16	18.14	0.90	24.57	10.55	0.99	6.52	2.76	0.99
	40	44.59	39.72	0.96	25.37	11.72	0.97	3.52	2.45	0.99

[a] $M(t) = M_0 + (t/k_1 + k_2 t)$, k_1 in h/(g water/g solids) and k_2 in g of solid/g water.

plasticizer/100 g soy protein, conditioned at 32% RH, had k_1 values of 33.09 and 170.41 h/(g water/g solids) for glycerol and sorbitol films respectively, while, in our work, cassava starch films (40 g plasticizer/100 g starch), conditioned at same conditions, presented lower k_1 values of 3.48 and 44.59 h/(g water/g solids) for glycerol and sorbitol films, respectively (Table 1). When k_2 parameters were compared, starch films also showed lower k_2 values in glycerol films (11.98 versus 20.45 g of solid/g water) but higher values in sorbitol films (39.72 versus 24.34 g of solid/g water). These data suggest that glycerol starch films adsorb more water and faster than glycerol protein films, while in sorbitol films, starch matrix adsorb faster but lower water content.

Glycerol and sorbitol plasticized films (at any plasticizer concentration) showed lower k_1 and k_2 values than those with, except when prepared with 20% plasticizer at 33% RH (Table 1). Similar observation was reported by Gaudin, Lourdin, Le Botlan, Ilari, and Colonna (1999), working with wheat starch–sorbitol films, and Myllarinen, Partanen, Sepalla, and Forsell (2002), working with amylose and amylopectin glycerol films. They related that at RH below 50%, the equilibrium water content for its films was lower in the presence of plasticizer than without it. According to Gaudin et al. (1999), sorbitol may be strongly bound with starch under low sorbitol contents (below 27), exerting an antiplasticization effect. Lourdin, Coignard, Bizot, and Colonna (1997) observed this effect in glycerol starch films at glycerol contents below 12%. In our work, we observed this phenomena only at 33% RH; this occurred probably because the plasticizer levels employed was very close or higher to the minimum of 27 and 12% reported to sorbitol and glycerol, respectively, or because the starch font was different. Cassava starch films had low amylose content (19%), and according to Myllarinen et al. (2002), amylopectin was more susceptible to plasticizing effect than amylose.

3.2. Moisture sorption isotherms of cassava starch films

The moisture sorption isotherms of cassava starch films are displayed in Fig. 2 and the GAB model parameters are shown in Table 2. Films without plasticizer presented the lowest monolayer value (0.035 g water/g solids), while the highest monolayer value (0.091 g water/g solids) was observed when 40 g glycerol/g starch was incorporated. The monolayer value indicates the maximum amount of water that can be adsorbed in a single layer per gram of dry film and it is a measure of number of sorbing sites (Strauss, Porcja, & Chen, 1991). The addition of plasticizer provides more active sites by exposing its hydrophilic hydroxyl groups in which the water molecules could be adsorbed.

Our results agreed with those of Cho and Rhee (2002), who found a similar trend for soy protein films plasticized with glycerol and sorbitol. Comparing monolayer values of starch films plasticized with 40 g plasticizer/100 g starch with protein films with 50 g plasticizer/100 g protein, starch films showed higher m_0 values in glycerol films (0.091 versus 0.085 g water/g solids), but lower values in sorbitol films (0.043 versus 0.054 g water/g solids), agreeing with kinetics results, that showed that glycerol starch films adsorb more water than glycerol protein films, while in sorbitol films, starch matrix adsorb lower water content.

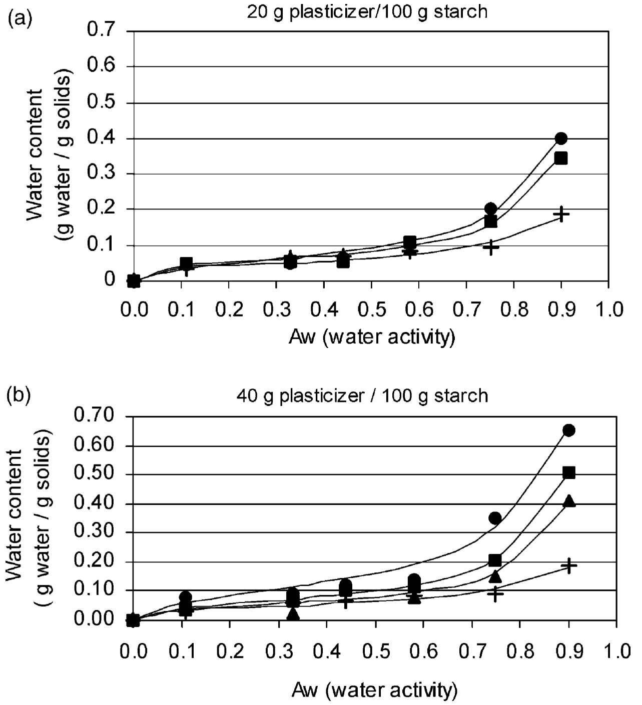

Fig. 2. Effect of plasticizer type and level on sorption isotherms for cassava starch films at 25 °C. •, films with glycerol; ■, films with glycerol:sorbitol; ▲, films with sorbitol and +, films without plasticizer. The lines were derived from GAB model.

The C parameter is related to the difference of the magnitude in the upper layers and in the monolayer (Timmermann, Chirife, & Iglesias, 2001); this parameter decreased with increased plasticizer concentration and glycerol-containing films showed the lower values (Table 2). This was according to findings of other authors, who also stressed that K parameter is effectively independent of composition (Coupland, Shaw, Monahan, O'Riordan, & Sullivan, 2000).

Table 2
GAB model[a] for cassava starch films at different plasticizer levels

Plasticizer	Plasticizer level (g/100 g starch)	m_0	C	K	r^2
Without	0	0.035	157.00	0.89	0.95
Glycerol	20	0.052	10.78	0.96	0.98
	40	0.091	11.99	0.96	0.98
Glycerol:	20	0.043	71.93	0.97	0.98
sorbitol	40	0.054	22.39	0.99	0.99
Sorbitol	20	0.043	238.00	0.97	0.99
	40	0.042	40.29	0.99	0.99

[a] $M = m_0CKa_w/(1-Ka_w)(1-Ka_w+CKa_w)$, where M is the equilibrium moisture content at a water activity (a_w), m_0 is the monolayer value (g water/g solids), and C and K are the constants.

In general, the moisture sorption isotherms for films showed a sigmoidal shape and were influenced by the concentration and by type of plasticizer (Fig. 2). Higher levels of plasticizer increased the films moisture affinity and these results could be attributed to the hydrophilicity of the plasticizers, which presented hydroxyl groups capable to interact with water by hydrogen bonds. In general, glycerol films showed higher capacity to adsorb water at all concentrations and at all RH conditions. Glycerol molecules are small and present high capacity to interact with starch chains, enhancing the molecular mobility and increasing free volume in the film matrix, besides, glycerol had a more hydrophilic character than sorbitol and these combined effects contributed to the higher water affinity of glycerol films (Sothornvit & Krochta, 2001).

3.3. Effects of RH and plasticizer on mechanical properties of cassava starch films

As shown in Fig. 3, a decrease in tensile stress was evidenced when RH increased. As shown before, the films stored at higher RH condition presented higher equilibrium moisture content, which exerted a plasticizing effect. Water exerted a plasticizing effect acting as a mobility enhancer; its low molecular weight leads a large increase in molecular mobility of amorphous and partially crystalline polymers due an increased free volume (van der Berg, 1991).

The plasticizer type and concentration affected the mechanical properties of cassava starch films; glycerol-containing films showed the lowest values for tensile stress (Fig. 3), due to its hygroscopic character that tends to provide additional water into the film matrix. Cuq, Gontard, Cuq, and Guilber (1997) stressed that plasticizers with lower molecular weight produced more film plasticization than the higher ones, at same mass basis, and suggested that plasticizer effect should be examined on a molar basis, i.e. when used at same mass basis, the number of moles of glycerol incorporated in the films were higher than the sorbitol ones, and this certainly is more important to the plasticizing effect.

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. Increasing plasticizer content resulted in films with lower Young's modulus and then more flexible (Fig. 4). The increase of flexibility with increasing plasticizer content in hydrophilic films have been reported previously (Mali et al., 2004; Mali et al.,

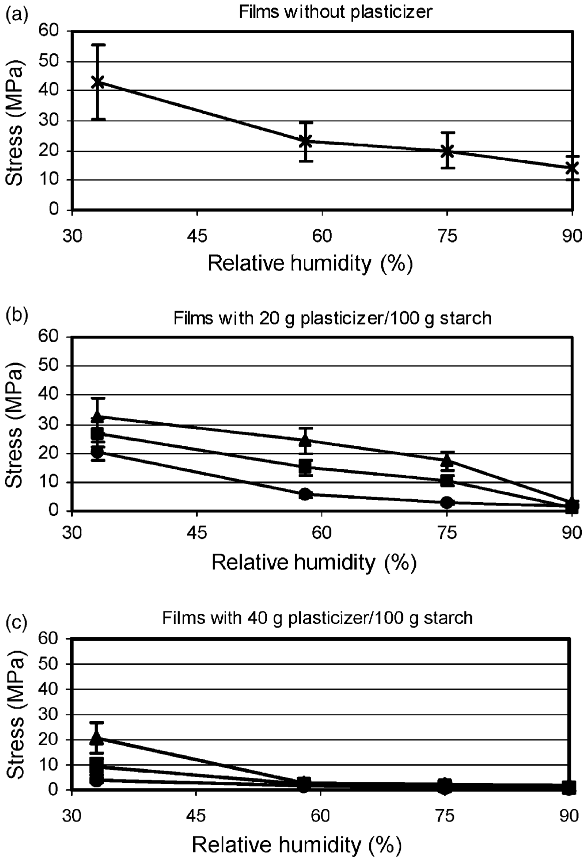

Fig. 3. Effects of relative humidity on tensile stress of cassava starch films. •, films with glycerol; ■, films with glycerol:sorbitol; ▲, films with sorbitol and ×, films without plasticizer.

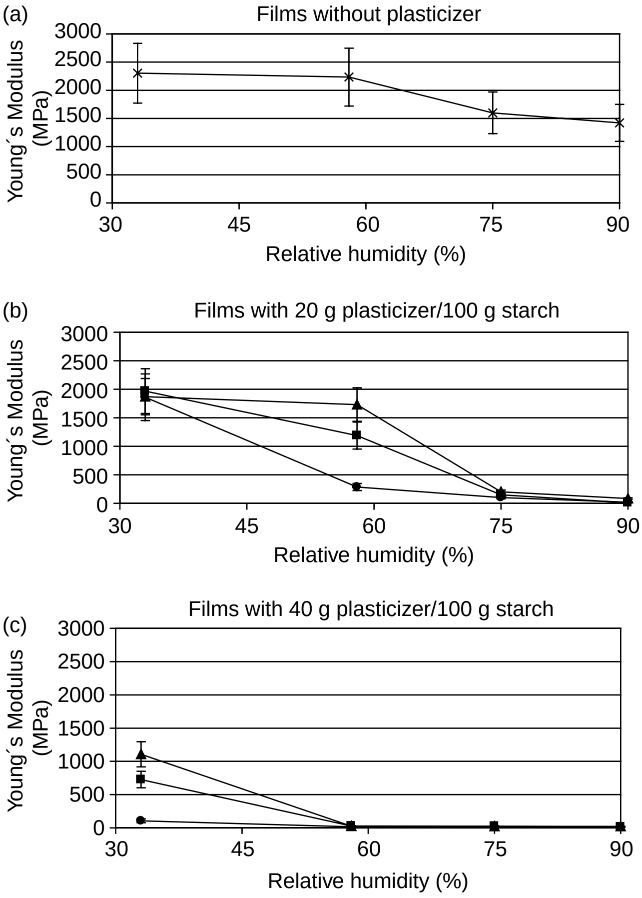

Fig. 4. Effects of relative humidity on Young's modulus of cassava starch films. •, films with glycerol; ■ = films with glycerol:sorbitol; ▲, films with sorbitol and ×, films without plasticizer.

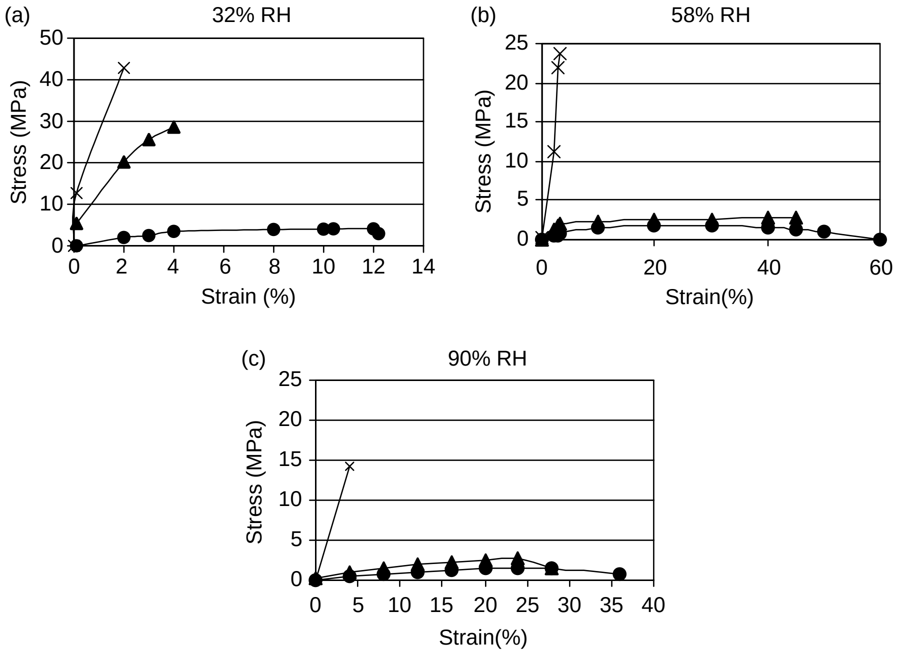

Fig. 5. Examples of stress–strain curves of unplasticized and plasticized (40 g plasticizer/100 g starch) cassava starch films. •, films with glycerol; ▲, films with sorbitol and ×, films without plasticizer.

2002; Parris et al., 1995; Sobral, Menegalli, Hubinger, & Roques, 2001). This behavior could be related to the structural modifications of starch network when plasticizer was incorporated, the matrix of the film become less dense and under stress movements of polymer chains were facilitated. The RH effect on Young's modulus could be observed in Fig. 4. By increasing RH, the values reached a minimum at 90% RH, and at higher levels of plasticizer (Fig. 4c) the effect of RH was more pronounced. At RH higher than 58%, the stress and Young's Modulus of all films containing 40 g plasticizer/100 g starch were similar, independent of type of added plasticizer.

The influence of plasticizer type under different relative humidity could also be explained looking at the primary tensile results (Fig. 5a–c) of unplasticized and plasticized films (40 g plasticizer/100 g starch). The tensile stress–strain curves show the more brittle character of the film prepared without plasticizer, under 32, 58 and 90% RH. At 32% RH, glycerol films were more flexible, while sorbitol films were brittle and strong. At RH higher than 58%, all films containing 40 g plasticizer/100 g starch presented very similar stress–strain curves, independent of plasticizer type, but glycerol films showed higher strain values, confirming its effectiveness as a plasticizer.

4. Conclusions

Hydrophilicity of plasticizer and its concentration were found to be important factors in determining the moisture affinity of cassava starch films. Peleg and GAB models were useful to fit moisture adsorption rates and sorption isotherm data, respectively. Glycerol films adsorbed faster and more water during its storage comparing to sorbitol films.

The presence of plasticizers resulted in lower stress and Young's Modulus values; glycerol films were more affected in its mechanical properties, indicating that glycerol exerted a more effective plasticization. Water was an effective plasticizer for starch films; thus, adsorbed water influenced and increased the plasticizing ability of added plasticizers. Although the plasticizers added at 40 g/100 g starch had conferred different mechanical characteristics to the films, these differences disappear with conditioning at RH higher than 58%.

Since moisture is adsorbed at different rates by different materials or by the same material under different humidity conditions, there was interesting to characterize the whole sorption process and not only the equilibrium conditions. Appropriate selection of plasticizer type and concentration will be helpful in controlling moisture content and moisture adsorption rate of a film, thereby improving the film stability under varying RH conditions.

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