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Physical, mechanical, and thermal properties of biodegradables films of rice starch

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ABSTRACT

It is known that the processing of rice produces large amounts of broken grains. As an alternative to increase the value of these broken grains there is the possibility to obtain rice starch, which transforms this raw material in a product with a greater industrial interest. Among the possible applications for starch, the use of biodegradable films is a feasible alternative in view of the partial synthetic polymers replacement. The aim of this study was to develop rice starch films containing different concentrations of plasticizer (40, 50 and 60 g glycerol/100 g starch) and analyze their physical, mechanical, and thermal properties, as well as color parameters. After all, the increase on glycerol content resulted in improvements on thickness, elongation, water vapor permeability, and mass loss by thermogravimetric analysis. Films with low glycerol concentrations showed better barrier properties. However, the tensile strengths decreased with the plasticizer content increment.

Keywords: starch rice, film, solubility, tensile strength, water vapor permeability.

INTRODUCTION

Rice is consumed in Brazil mostly under the form of whole grains, dehulled and polished, as opposed to what happens for wheat and corn, which are transformed in other products for consumption, such as flours (Castro et al., 1999). The development of more sophisticated products using rice as raw material is inconsistent with the purchasing power of the majority of the world's population, traditionally consumer of rice. However, the use of its co-products becomes feasible because the rice processing yields approximately 14% of broken grains (Dias et al., 2010). These broken grains are used for animal feeding or at markets with low commercial values.

Starch extraction is an alternative to add value to broken rice grains transforming this raw material in a product with greater industrial interest. Rice varieties contain starches with different characteristics allowing for multiple applications, such as preparation of pasta (Cham and Suwannaporn, 2010; Hormdok and Noomhorm, 2007), gluten free breads (Clerici, Airoldi and El-Dash, 2009; López et al., 2004; Ding et. al. 2005; Kadan et al, 2001), mixtures for the preparation of snacks (Suksomboon et al., 2011) and also of biodegradable films (Bourtoom, 2008; Bourtoom and Chinnan, 2008; Rachtanapun and Tongdeesoontorn, 2009; Dias et al., 2010; Dias et al., 2011).

The exponential growth of the population, mainly urban, in addition to industrial development and new consumption patterns has led to an increase on generation of plastic packaging such that it may cause environmental problems. Therefore, there is great interest in developing biodegradable films to be used in packaging. These films can be used as food packaging, since they promote reduction of moisture loss, restriction of oxygen loss, and reduction of lipid migration. Moreover, it provides physical protection and an alternative to commercial packaging materials (Bourtoom, 2009).

Starch is the main reserve component in plants and the most important raw material for many industrial processes (Smith et al., 1997). It is also the main component of rice grains and functional ingredient in food preparations (Sasaki et al., 2009). Starch mixed with other compounds has been used as raw material for the production of biodegradable films, mainly because this is a material of low cost, primarily for its large availability. In addition, it is harmless to the environment, since once discarded it is rapidly metabolized by soil organism (Dias et al., 2011; Rosa et al., 2001). The production of biodegradable and/or edible films made from starch adds value to low value raw materials and fulfills a role in food preservation (Avérous et al., 2004).

The addition of plasticizing agents is essential for preparation of films. They are composed of small molecules of low volatility. When added to a starch based material, they interact with the starch chains and improve the molecular mobility and thus the flexibility of the resultant films. Moreover, the plasticizers should be compatible, i.e., they must exhibit the same hydrophilic or hydrophobic character of polymer. The plasticizers widely used in starch films are polyols such as glycerol and sorbitol (Hu et al., 2009; Myllarinen et al., 2002). According to Lourdin et al. (1997) efficient plasticizers promote favorable interactions through hydrogen bonds with starch.

Films applications are conditioned by characteristics such as cost, availability, functional attributes of the macromolecule, barrier, mechanical and sensory properties of the films (Baldwin and Carriedo, 1994). The aim of this study was to evaluate the effect of glycerol concentration on the physical, mechanical, barrier and thermal properties of biodegradable films from rice starch.

MATERIAL AND METHODS

Starch Extraction

Rice grains of cultivar IRGA 417 with amylose content of 32% were used. The extraction was based on the method described by Zavareze et al. (2010) with some modifications. Rice starch was isolated with 0.1% NaOH as described by Wang and Wang (2004). Rice flour was soaked in 0.1% NaOH in a 1:2 (w/v) ratio for 18 h at room temperature (25 ± 2 °C). Then it was blended, passed through a 63 µm screen and centrifuged at 1200 *g* for 5 min. The soft-top layer was carefully removed, and the underlying starch layer was re-slurried. The starch layer was then washed twice with 0.1% NaOH and centrifuged. The starch layer was washed with distilled water and centrifuged. The starch was then re-slurried and neutralized with 1.0 M HCl to a pH of 6.5 and centrifuged. The neutralized starch was washed with distilled water three times and dried at 40°C to 10 ± 0.5% moisture.

Film Preparation

Films were made following the casting technique, according to Shimazu et al. (2007). The native starch was homogenized in distilled water at a rate of 4 g of starch / 100 mL and then the plasticizer (glycerol) was added at concentrations of 40, 50 and 60 g glycerol/100 g starch. The solutions were heated in a glass reactor with constant stirring until reaching 85 ºC and this temperature was maintained for one hour. The film-forming solutions were poured into acrylic plates (90 mm diameter) and dried at 40 °C for 16 h in an oven with air circulation (Greenhouse series 400, Nova Ética). Samples of dried films were conditioned at 25 °C in a desiccator for two days using silica as desiccant.

Determining film thickness

Film thickness was determined on three samples using a micrometer (model INSIZE) to the nearest 0.001 mm at 8 random positions around the film, and the average values were used in the calculations. Results were expressed in mm (Monterrey and Sobral, 1999).

Water Solubility

Solubility was calculated as the percentage of dry matter of the solubilised film after immersion in water for 24 h at 25 °C (Gontard et al., 1994). Film discs (2 cm in diameter) were cut, weighed, immersed in 50 mL of distilled water and stirred at 175 rpm (shaker, Certomat® MO, B. Braum, Biotech, International). The amount of dry matter in the initial and final samples was determined by drying the samples at 105 °C for 24 h. The solubility was calculated using Equation 1 as follows:

$$SW(\%) = \frac{(W_0 - W)}{W} \times 100 \quad \text{Eq. (1)}$$

where SW is the solubility in water; and W_0 and W are the dry sample weights before and after the test, respectively.

Mechanical properties

The tensile strength and percentage of elongation at break was evaluated in a tensile test and measured using a texture analyser (TA.XTplus, Stable Micro Systems) based on the ASTM D-882-91 standard method (2000). Films were cut in strips (25 mm x 85 mm) and conditioned for two days in a desiccator as previously described. Force and distance were recorded during the extension of the strips at 0.8 mm/s up to the breaking point. Tensile strength was calculated by dividing the maximum force by the film cross-section. The tensile strength and percentage elongation were calculated using Equations 2 and 3, respectively, as follows:

$$TS = \frac{F_m}{A} \quad \text{Eq. (2)}$$

$$E = \frac{d_r - d_o}{d_0} x100 \quad \text{Eq. (3)}$$

where TS is the tensile strength (MPa), F_m is the maximum force (N), A is the area of film cross-section (thickness x width; m^2), E is the elongation (%), d_0 is the distance onset of separation (cm) and d_r is the distance of rupture (cm).

Water vapor permeability

Water vapor permeability (WVP) tests were conducted using the ASTM E96-00 standard method (ASTM, 2000). Each film sample was used to seal the circular opening of a permeation cell containing anhydrous calcium chloride (2% RH). These cells were placed on desiccators with a saturated sodium chloride solution (75% RH) and kept at 25 °C. After the samples reached steadystate conditions, cell weight was measured at 48 h. The WVP was calculated using Equation 4 as follows:

$$WVP = \frac{\Delta W}{t}\frac{X}{A\Delta P} \quad \text{Eq. (4)}$$

where *WVP* is the water vapor permeability (g.mm/KPa.day.m^2), *ΔW* is the weight gain by desiccant (g), *X* is the film thickness (mm), *t* is the incubation time period (days), *A* is the area of the exposed film surface (m^2) and *ΔP* is the difference of partial pressure (KPa).

Thermal Analysis

Thermogravimetric analysis the films was performed on thermogravimetric analyzer (Shimadzu model DTG 60, Osaka, Japan) at a rate between 25 and 600 °C with a nitrogen flow of 50 mL/min of N_2 according to the parameters proposed by Zhong et al (2013).

Color and opacity

The color of the films was determined with a colorimeter (Minolta, CR 400, Osaka, Japan) operating with D65 (daylight) and using the CIELab color parameters (Hunterlab, 1997). The color and CIELab color difference meter identifies color in the following three attributes: L* (100 = white; and 0 = black), a* (positive = red; and negative = green), and b* (positive = yellow; and negative = blue). The instrument was calibrated using a white standard color calibration plate. Opacity (Y) was calculated from the relationship between the opacity of the film superimposed on the black standard (Y_{black}) and that of the film superposed on the white standard (Y_{white}) according to Equation 5 as follows:

$$Y = \frac{Y_{black}}{Y_{white}} \times 100 \quad \text{Eq. (5)}$$

The color difference (ΔE*) as compared to a white standard tile of the colorimeter was calculated using Equation 6, as follows:

$$\Delta E^* = \sqrt{(L^* - L_s^*) + (a^* - a_s^*) + (b^* - b_s^*)} \quad \text{Eq. (6)}$$

where *L**, *a** and *b** are the color attributes of the film samples, and L_s^*, a_s^* and b_s^* are the color parameters of the white standard tile.

Statistical analysis

Analyses of variance (ANOVA) were performed using the Statistica 7.0 V (Startsoft). Significant differences between means were identified by Tukey test ($p < 0.05$).

RESULTS AND DISCUSSION

Thickness, tensile strength and elongation of rice starch films prepared with different glycerol concentrations are shown in Table 1. The thickness of the films ranged from 0.142 to 0.164 mm (Table 1) and increased as the concentration of glycerol was increased. According to Embuscado and Huber (2009), biodegradable films usually have thicknesses of less than 0.300 mm, which is an important physical characteristic, since its use as packaging must consider the type, volume and weight of the food that will be stored.

Film thickness is an important characteristic that allows indicates the mechanical strength and barrier properties of the material to gases and to water vapor. The determination of thickness is also important to assess the homogeneity of film. Variations in thickness cause problems in the film's mechanical performance and variations in barrier properties (Cetea, 1996).

Films must have sufficient mechanical strength to ensure its integrity when used as packaging. The mechanical properties of tensile strength and elongation of rice starch films are presented in Table 1. Films with 50% and 60% glycerol had lower tensile strength (0.55 and 0.60

MPa), respectively, when compared with films of lower plasticizer concentration (1.13 MPa). Mechanical properties of starch films are important, since these materials must be resistant to breakage and abrasion, to protect and strengthen the structure of the food, and also should be flexible to adapt to the possible deformations without breaking (Sobral, 2000). Myllarinen et al. (2002) reported that glycerol and starch polymers presented a strong interaction, and at low glycerol content (10%) this interaction resulted in a more brittle structure.

Elongation is a measure of film flexibility and may be considered as a characteristic that defines the ability of the film to deform before failure. The elongation of the films with 50% and 60% glycerol was significantly higher than that of films with 40% of plasticizer (Table 1). This behavior is due to ability of glycerol to reduce interactions between polymer chains, thereby decreasing the film strength and increasing their flexibility (Sothornvit and Krochta, 2000). Glycerol concentration is an important factor capable of altering the profile of mechanical properties of starch films and with increasing plasticizer content decreases the resistance and increases the flexibility (Mali et al., 2004).

Water vapor permeability is the rate of water vapor transmission through unit area of the material of a certain thickness, induced by the difference in vapor pressure between two specific surfaces under defined temperature conditions (ASTM E96-00, 2000). Water vapor permeability increased significantly with the addition of 60% glycerol (Table 2). According to McHugh and Krochta (1994), the increased glycerol content tends to increase the water vapor permeability, since this it binds to the biopolymer molecules increasing the density and decreasing the mobility of the molecules, facilitating permeation.

Different concentrations of glycerol did not affect the solubility of the films. Solubility values were lower than the results obtained by Mehyar & Han (2004) who studied rice starch films and found 44% of water solubility. Plasticizers promote a decrease in intermolecular forces and the network becomes less dense. Thus improving flexibility and extensibility of the films followed by a decrease in mechanical strength, and glass transition temperature and gas and water vapor barrier. This fact can be explained by the hygroscopicity of plasticizer and its action in breaking the network, moistening the starch-plasticizer interactions, and consequently decreasing the cohesion of the molecule, in addition to increasing the free volume (Sothornvi and Krochta, 2000; Paschoalick et al, 2003; Mota, 2009)

Table 1. Thickness, tensile strength and elongation of rice starch films prepared with different glycerol concentrations

Glycerol (%) [a]	**Thickness (mm)**	**Tensile strength (MPa)**	**Elongation (%)**
40	0.142 ± 0,07 [b]	1.13 ± 0,13 [a]	60.7 ± 9,48[b]
50	0.152 ± 0,09[ab]	0.55 ± 0,04[b]	100.4 ± 4,09[a]
60	0.164 ±0,11 [a]	0.60 ± 0,07 [b]	116.6 ± 8,75[a]

[a] Different letters in the same column differ statistically ($p < 0.05$).

Weight loss, verified by thermogravimetry (Table 2), increased with the addition of glycerol in the composition of the films. This behavior is due to the plasticizer being a small molecule with low volatility. When it is added to the filmogenic solution, it modifies the molecular organization of the starch network, increasing the free volume in the starch molecule (Sothornvi and Krochta, 2000; Paschoalick et al., 2003; Mota, 2009).

Color and transparency (opacity) are relevant characteristics of films for use in packaging. Color is an important parameter for the characterization of the films, since it is associated with the raw material used to make the products (Bertuzzi et al., 2007). For a good visual presentation of products it is desirable for the material to have high gloss and high transparency, though these are not limiting factors of use. The color and opacity of the films of rice starch prepared with different plasticizer concentrations are presented in Table 3. Films with different concentrations of glycerol did not differ significantly ($p < 0.05$) for the parameters L *, a * and b * (Table 3). However, incorporation of 60% glycerol in starch films reduced opacity when compared to those films produced with 40% of the plasticizer (Table 3).

Table 2. Water vapor permeability (WVP), solubility and thermogravimetric analysis (mass loss) of rice starch film with different plasticizer concentrations

Glycerol (%) [a]	WVP 48 h (g.mm/m^2.day.kPa)	Solubility (%)	Mass loss (%)
40	5.26 ± 1,48 [b]	18.14± 1,11 [a]	82.5
50	5.57 ± 1,60 [b]	19.67 ± 0,88 [a]	83.9
60	9.22 ± 1,39 [a]	20.13 ± 0,53 [a]	86.1

[a] Different letters in the same column differ statistically ($p <0.05$).

The rice starch film prepared with 50% of glycerol was significantly different to the others for color variation (ΔE) and opacity (Table 3). Bertuzzi et al. (2007) observed that the use of plasticizer leaves the network structure more flexible and the films more opaque, with the opacity increasing with the addition of plasticizer. They also found that for concentrations above 15% of glycerol there are structural changes, increasing mobility of the chains and decreasing the opacity.

Table 3. Analysis of color and opacity of rice starch films with different plasticizer concentrations

Glycerol (%) [a]	L*	a*	b*	ΔE [b]	Opacity (%)
40	97.32 ± 0,16 [a]	0.20 ± 0,03 [a]	1.77 ± 0,09 [a]	0.82 ± 0,16 [b]	10.26 ± 0,02 [a]
50	97.10 ± 0,22 [a]	0.20 ± 0,01 [a]	1.84 ± 0,10 [a]	1.09 ± 0,23 [a]	10.79 ± 1,01 [ab]
60	97.27 ± 0,13 [a]	0.22 ± 0,03 [a]	1.74 ± 0,05 [a]	0.82 ± 0,10 [b]	9.27 ± 1,20 [b]

[a] Different letters in the same column differ statistically ($p <0.05$).
[b] ΔE: Color difference

CONCLUSIONS

The amount of glycerol did not affect the solubility and color of the rice starch films and the films had high transparency and brightness. The films made with lower glycerol concentrations showed better properties due to the lower thickness and lower water vapor permeability, and also a decrease on the weight loss.

The preparation of rice starch films is a new alternative to the use of this raw material, however further studies should be made with the aim of improving the features that are still precarious in the films.

The development of biodegradable films can provide benefits such as the development of sustainable technologies, conservation of fossil resources, encouraging greater use of renewable resources and use of by-products and waste from food production.

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