



## Structural and mechanical properties of edible films made from native and modified cush-cush yam and cassava starch



Tomy J. Gutiérrez <sup>a, \*</sup>, María Soledad Tapia <sup>a</sup>, Elevina Pérez <sup>a</sup>, Lucía Famá <sup>b</sup>

<sup>a</sup> Instituto de Ciencia y Tecnología de Alimentos, Facultad de Ciencias, Universidad Central de Venezuela, Apartado 47097, Caracas 1041-A, Venezuela

<sup>b</sup> LP&MC, IFIBA-CONICET, Facultad de Ciencias Exactas y Naturales, Universidad de Buenos Aires, Ciudad Universitaria, Pab. 1, Buenos Aires, C1428EHA, Argentina

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### ABSTRACT

Several different hydrocolloids, such as starch, have been proposed as suitable base materials (matrices) for edible films in food packaging. Edible films from native and modified starch plasticized with glycerol were developed. Starches were obtained from dark cush-cush yam (*Dioscorea trifida*) and cassava (*Manihot esculenta* C.) from Venezuela, and chemically modified by cross-linking with sodium trimetaphosphate. The uniaxial tensile, microstructural and barrier properties of the films were then evaluated to determine their potential as a replacement for existing synthetic materials used in the food industry. The structure of the materials showed that the gelatinization process of cush-cush yam films was poorer than that of cassava. The glycerol–starch interaction (glycerol–amylose) was stronger in the films composed of modified starches and was more marked in cassava based films. All the films studied exhibited promising mechanical properties, with those derived from cush-cush yams showing the highest Young's modulus and resistivity values. Cassava based edible films and films derived from modified starch from both sources showed maximum flexibility, reinforcing the idea that the glycerol–starch interactions are stronger in these materials. Crosslinked films tended to be more permeable to water vapor due to their hydrophilic characteristics. The properties observed in these biodegradable materials highlight their potential as food packaging materials, thus enabling the replacement of synthetic materials that contaminate the environment.

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### 1. Introduction

Starch stands out as a potential biopolymer matrix for the manufacture of edible plastics due to its ubiquitous, cheap and biodegradable nature. Tropical roots and tubers are valuable sources of amylaceous materials from which starch-based edible films and coatings could be produced. Several studies have focused on the properties and manufacture of starch-based films derived from various botanical sources, showing that roots and tubers are promissory for these purposes (González & Pérez, 2003; Pérez, Borneo, Melito, & Tovar, 1999; Pérez, Gibert, Rolland-Sabaté, et al., 2011; Pérez, Gibert, Sabate, et al., 2011; Pérez, Schultz, & Pacheco, 2005; Pérez, Segovia, Tapia, & Schroeder, 2012; Tapia et al., 2012). In particular, white and purple *Dioscorea trifida* has been reported

as a new waxy yam starch with promising properties (Pérez, Gibert, Sabate, et al., 2011). However, the films made from this starch should be investigated more thoroughly due to its waxy nature (Tapia et al., 2012). Cassava starch has been extensively studied for its renewability, low cost, biodegradability and wide availability (Matos & Pérez, 2003; Pérez, Breene, & Bahanasey, 1998; Sívoli, Pérez, Rodríguez, De Abrisqueta, & Raymúndez, 2005). In recent years native starches from tropical crops have been used to prepare edible films (García, Martino, & Zaritzky, 2000) as coatings for the protection and enhancement of fruit products (Hernández, Emaldi, & Tovar, 2008; Rojas-Graü, Tapia, & Martín-Belloso, 2007). The amylaceous components are naturally packed in crystalline regions to form the granular semi-crystalline structure of the starch. As the waxy starches contain low amylose within the starch granule, this produces a higher relationship of crystallinity between amylopectin and amylose, easily producing a plastic structure.

The modification of starch by cross linking (Matos & Pérez, 2003; Sívoli et al., 2005) can improve the functional properties

\* Corresponding author. Tel.: +58 212 7534403; fax: +58 212 7533971.

E-mail addresses: [tomy.gutierrez@ciens.ucv.ve](mailto:tomy.gutierrez@ciens.ucv.ve), [tomy\\_gutierrez@yahoo.es](mailto:tomy_gutierrez@yahoo.es) (T.J. Gutiérrez).

not only of starch, but also of edible starch-based coatings, films and foams. Starch can be chemically modified by cross-linking with sodium trimetaphosphate which improves its functional properties leading to higher paste clarity and consistency, stability during freeze–thaw cycles and the ability to form an emulsion due to its polar nature (Lim & Seib, 1993; Whistler & Paschall, 1967; Wurzburg, 1986). Similarly, cassava starch phosphated has very high viscosity and low cohesiveness (Moorthy, 1994). Despite the large amount of research done on starch based films the ultrastructure and the mechanical properties of cross-linked thermoplastic starch materials has been little studied (Jiménez, Fabra, Talens, & Chiralt, 2012).

The aim of this study was to evaluate the ultrastructure, uniaxial tensile strength, water vapor permeability and color properties of edible films prepared from native and cross-linked cush-cush yam and cassava starches.

## 2. Experimental

### 2.1. Materials

Native starch from dark purple cush-cush yam (*D. trifida*) was obtained from a variety from the Venezuelan Amazon ( $12 \pm 3\%$  apparent amylose) and cassava starch by extracting the starch from a variety of cassava (*Manihot esculenta* C.) ( $21 \pm 3\%$  apparent amylose) on sale at a local market in Caracas, Venezuela (Gutiérrez, Pérez, Guzmán, Tapia, & Famá, 2014). The extraction of the starch from the cush-cush yam and cassava tubers was carried out using the methodology described by Pérez, Bahnassay, and Breene (1993), obtaining in both cases a yield of approximately 30% (Gutiérrez, Pérez, et al., 2014). Modified starches were prepared from native starches by cross-linking method (phosphating of the starches). The apparent amylose content of the modified dark purple cush-cush yam starch and the cassava starch was  $\sim 11\%$  and  $\sim 22\%$ , respectively (Gutiérrez, Pérez, et al., 2014). The degrees of substitution (DS) of the starches were:  $0.0006 \pm 0.0002\%$  and  $0.017 \pm 0.009\%$  for native and modified dark purple cush-cush yam, respectively, and  $0.0015 \pm 0.0002\%$  and  $0.008 \pm 0.001\%$  for native and modified cassava, respectively (Gutiérrez, Pérez, et al., 2014). Glycerol from Prolabo, Sweden, was employed as plasticizer in the formation of the films.

### 2.2. Preparation of the modified starch

Phosphate-modified starch was prepared using sodium trimetaphosphate, following the methodology described by Kerr & Cleveland (1959), modified by Lim and Seib (1993). The maximum concentration of the modifying agent allowed by the FDA for starches intended for the food industry (3% W/W of sodium trimetaphosphate with respect to the weight of the starch) was used.

### 2.3. Film formation

Edible films were prepared from a film forming solution (FFS) made by mixing 2% W/V of starch and 1.9% W/V of glycerol in distilled water. The solution was then heated in a water bath with constant stirring at  $90^\circ\text{C}$  for 30 min to ensure starch gelatinization (Hernández, 2006). The gel was then degassed by applying a vacuum for 30 min, poured into stainless steel trays  $40 \times 30$  cm, and dried in a Mitchell dehydrator (Model 645 159) for 24 h at  $45^\circ\text{C}$ . The resulting thermoplastic starch films: native cush-cush yam (TPS-NY), phosphated cush-cush yam (TPS-PY), native cassava (TPS-NC) and phosphated cassava (TPS-PC) were then carefully removed from the casting molds. Before characterization, the films were conditioned at  $\sim 57\%$  relative humidity (RH) for a week.

## 2.4. Characterization

### 2.4.1. Light microscopy

Small pieces ( $2\text{ cm} \times 1\text{ cm}$ ) of each film were mounted onto glass slides, examined using an optical microscope (Olympus BX60M, Japan) at  $50\times$  and photographed with a video camera imaging system (Olympus IMAGE RS). At least three microphotographs of each system were taken.

### 2.4.2. Scanning electron microscopy (SEM)

The films were cryofractured by immersion in liquid nitrogen, mounted on bronze stubs and sputter coated with a thin layer of gold for 35 s. The fracture surface of each material was then analyzed using a Philips XL series 30 (Holanda) scanning electron microscopy (SEM).

### 2.4.3. Determination of film thickness

The thickness ( $e$ ) of the films was determined following the methodology described by Rojas-Graü, Raybaudi-Massilia, et al. (2007) and Rojas-Graü, Tapia, Rodríguez, Carmona, and Martín-Belloso (2007). A digital micrometer (Micromaster<sup>®</sup>) with an accuracy of 0.001 mm was used. For each film a total of 18 measurements were taken at several randomly selected points and the mean thickness calculated. The results were used for the determination of water vapor permeability and tensile properties.

### 2.4.4. Water vapor permeability (WVP)

Water vapor permeability was measured following ASTM E96-00 (1999) and the correction method described by Gennadios, Weller, and Gooding (1994). Circular acrylic cells containing pieces of film (exposed area  $\sim 15.2\text{ cm}^2$ ) were introduced into desiccators at ambient temperature with a relative humidity of  $\sim 50\%$ .

WVP values were calculated as:

$$\text{WVP} = \frac{G \times e}{S \times \text{RH} \times t \times A} \quad (1)$$

where  $G$  is the mass gained,  $e$  the thickness of the film,  $S$  the saturated vapor pressure at  $25^\circ\text{C}$ , RH the relative humidity,  $t$  time, and  $A$  the exposed area of each sample.

All assays were performed in triplicate, reporting the average and standard error in each case.

### 2.4.5. Color

The following color parameters of the films:  $L^*$ , (where  $L^* = 0$  indicates black and  $L^* = 100$  white),  $a^*$  (position between red and green) where negative values indicate green while positive values indicate magenta, and  $b^*$  (position between yellow and blue) where negative values indicate blue and positive values indicate yellow, were measured according to the standard test method (ASTM D-1925, 1995) using a Macbeth<sup>®</sup> colorimeter (Color-Eye 2445 model, illuminant D65 and  $10^\circ$  observer) standardized with a white reference plate ( $L^* = 93.52$ ,  $a^* = -0.81$  and  $b^* = 1.58$ ). Color differences ( $\Delta E$ ), as measured by the magnitude of the resultant vector of the three components: brightness difference,  $\Delta L$ , red-green chromaticity difference,  $\Delta a$ , and yellow-blue chromaticity difference,  $\Delta b$  (Valencia Rodríguez, 2001), were calculated by the following equation:

$$\Delta E = \sqrt{\Delta a^2 + \Delta b^2 + \Delta L^2} \quad (2)$$

where  $\Delta a = a_i - a$ ,  $\Delta b = b_i - b$ ,  $y$  and  $\Delta L = L_i - L$ . The index  $i$  is the reference value of each parameter.

The yellowness index (YI) which, as its name suggests, determines the degree of yellowness of a substance, was calculated

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