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Optimization of physical properties of xanthan gum/tapioca starch edible matrices containing potassium sorbate and evaluation of its antimicrobial effectiveness

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ABSTRACT

Response surface methodology was applied to study the effect of different levels of tapioca starch (TS) and xanthan gum (XG) on physical properties of edible films supporting potassium sorbate (KS) with the goal of contributing to the development of edible matrices with controlled release of the antimicrobial. Mechanical properties, water vapor permeability (WVP), solubility in water (SW) and color attributes were evaluated on TS–XG based films. XG addition produced an increase of Young modulus (YM), stress at break (σ_b) and SW. It also raised the yellow index (YI) values and decreased the strain at break (ε_b). Edible film formulation was optimized with the goal of maximizing YM and ε_b and minimizing SW and YI. The film with the selected formulation resulted an effective antimicrobial barrier against *Zygosaccharomyces bailii* external contamination and its sorptional behavior was highly influenced by XG presence. It can be concluded that developed matrices could act as an effective active film with potential applications for food preservation.

1. Introduction

Edible films and coatings are one of the emerging strategies for food quality optimization. Their usefulness is based on the capacity of optimizing global quality, extending shelf life and, possibly, improving the economic efficiency of packaging materials (Rojas-Graü, Soliva-Fortuny, & Martin-Belloso, 2009).

These matrices can be used to cover food surfaces, compartmentalize ingredients, form a gas barrier or constitute wraps. They can be used as carriers of additives or to improve appearance and handling (Campos, Gerschenson, & Flores, 2011). In general, these films have high water solubility and poor WVP. To solve this shortcoming, the blending of different biopolymers (Xu, Kim, Hanna, & Nag, 2005) or the addition of hydrophobic materials such as oils or waxes (Ayranci & Tunc, 2003) has been proposed.

Tapioca starch ability as a former of edible film matrices has been reported (Flores, Famá, Rojas, Goyanes, & Gerschenson, 2007).

Xanthan gum (XG) is a high molecular weight extracellular polysaccharide produced by the bacterium *Xanthomonas campestris*. Flores, Costa, Yamashita, Gerschenson, and Grossmann (2010) informed that its incorporation to starch-based films obtained through extrusion, affected mechanical properties and water sorption of the films.

The KS is usually employed in food preservation because of its GRAS (Generally Recognized as Safe) status and high solubility in water. Films and coatings containing sorbates (KS and sorbic acid) have been developed to inhibit the growth of yeasts in foods systems (Campos et al., 2011). Previous research of the authors showed that sorbates changed the physical properties of films based on slurries containing 5 g/100 g of tapioca starch (Flores, Famá, et al., 2007) or a blend of starch and chitosan (Vásconez, Flores, Campos, Alvarado, & Gerschenson, 2009). Cagri, Ustunol, and Ryser (2001) and Shen, Wu, Chen, and Zhao (2010) informed the same trend for films based on whey proteins and sweet potato starch, respectively.

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The application of antimicrobial edible films might help to obtain a localized functional effect at the surface of the food product (Flores et al., 2010) or to produce the gradual release of the antimicrobial through the control of its diffusion to the food or to protect the additive from the interaction with other food components or from ambient conditions which could promote its destruction or inactivation (Rojas-Graü et al., 2009). It is important to remark that film forming conditions and film composition affect additive migration and, as a consequence, its effectiveness (Flores, Conte, Campos, Gerschenson, & Del Nobile, 2007).

Response surface methodology (RSM) is a useful collection of mathematical and statistical techniques for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response (Montgomery, 2005).

The objectives of this study were:

- to evaluate through an RSM, the influence of TS and XG levels on the physical properties of TS-based edible films, with the purpose of optimizing the formulation.
- to determine the antimicrobial behavior and water vapor sorption of the optimum film,

for contributing to the knowledge of biopolymer matrices behavior as carriers of additives in order to control the release of antimicrobials on the surface of foods.

2. Material and methods

2.1. Preparation of edible films

XG (Degussa S.A., Argentina) was dissolved in water under stirring at room temperature for 30 min. A suspension of TS (Bernesa, Argentina) in a glycerol (Sintorgan, Argentina) – KS (Sigma, USA) aqueous solution was added to the gum, under stirring, until total homogenization. Heating of hydrocolloid systems was performed (1.5 °C/min) on a hotplate under stirring to completely gelatinize the starch (final temperature: 80–82 °C). The XG and TS final contents varied according to Table 1. Glycerol and KS final concentrations were maintained at 2.5 and 0.3 g/100 g slurry respectively for all systems through the addition of 7.5 g of glycerol and 0.9 g of KS into a system of 300 g final weight. Vacuum was applied to remove air from the solutions.

Casting was used for edible film obtention: 20 g of film forming solutions were dispensed on Petri dishes (9 cm diameter) and dried in a controlled temperature chamber (25 °C) for 48 h. Film was separated and equilibrated to a final water activity (a_w) of 0.57 (25 °C) over NaBr saturated solution.

2.2. Mechanical properties of films

Traction tests were performed according to ASTM D882-10 (2010) with slight modifications. Tested filmstrips (6 mm × 60 mm) were cut and mounted between pneumatic grips. Initial grip separation and crosshead speed were 20 mm and 0.8 mm/s respectively. The stress σ (F/A, being F the force and A the area of the specimen; MPa) and the strain ε (H/L_0, being H the crosshead displacement occurred and L_0 the initial effective length of the sample; %) were recorded using a universal testing machine (Instron 3345, USA). The stress and the strain at break (σ_b, ε_b) were evaluated. For strains lower than 10%, the stress–strain curves were fitted to a linear model and the YM (MPa) was evaluated from the slope. Nine specimens were tested for each sample.

Table 1
Independent variables tapioca starch (TS), xanthan gum (XG) and their levels for the Central Composite Design.

Variable levels	Independent variables	
	TS (g/100 g slurry)	XG (g/100 g slurry)
−2	4.00	0.00
−1	4.25	0.25
0	4.50	0.50
+1	4.75	0.75
+2	5.00	1.00

2.3. Solubility in water

Solubility is defined (Gontard, Guilbert, & Cuq, 1992) as the percentage of dry matter solubilized after 24 h of film immersion in distilled water with respect to initial dry matter. The initial dry matter was determined by drying 2 cm diameter disks in a vacuum oven at 100 °C during 24 h. Other disks were cut, weighed and immersed in 50 mL of distilled water, with stirring, during 24 h at 25 °C. Not solubilized films were taken out and dried (100 °C, 24 h) to determine the final weight of dry matter. Three specimens were tested for each sample.

2.4. Color evaluation

Film disks were rested on white background standard. Measurements were performed in a Minolta colorimeter (Minolta CM-508d, Japan) using an aperture of 1.5 cm-diameter. The color Hunter Lab parameters and the yellow index, YI (ASTM E1925, 1988), were determined in at least five positions randomly selected for each sample. Calculations were made for D65 illuminant and 2° observer. Three specimens were tested for each sample.

2.5. Water vapor permeability

WVP was determined gravimetrically at 25 °C according to ASTM E96-00 (2000) procedure with modifications. The acrylic permeation cells contained $CaCl_2$ (0% RH). Film was located between the cell and its ring cover. The covered cell was placed in a temperature and RH controlled chamber (Ibertest, España) at 25 °C and an RH of 70%. Changes in weight of the cell were recorded and used for WVP calculation. The film thickness used for calculations was measured using a thickness gauge (Mitutoyo, Japan) with a precision of 0.001 mm. All tests were conducted in duplicate.

2.6. Moisture sorption isotherm

Aliquots of 0.3–0.4 g of films based on slurries containing TS 4.75 g and XG 0.25 g/100 g slurry or TS 5 g/100 g slurry and both with 2.5 g of glycerol and 0.3 g of KS per 100 g of slurry, were equilibrated at 25 °C in desiccators containing $CaCl_2$ (0% RH). The moisture adsorption isotherms of the films were determined in triplicate, at 25 °C, with the gravimetric method (Mathlouthi, 2001). Ten different relative humidity conditions (11.3, 32.8, 43.1, 57.6, 75.3, 84.3, 90.1 and 97.3% R.H.) obtained by using saturated salt solutions (Greenspan, 1977) were evaluated. The sample weights were measured to the nearest 0.0001 g at 25 °C, until the films reached equilibrium. The equilibrium moisture content was determined by drying samples in a vacuum oven at 70 °C until reaching a constant weight. The experiment was performed in triplicate.

The experimental moisture sorption values were fitted to the Oswin empirical model: