



Effect of oil lamination between plasticized starch layers on film properties



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ABSTRACT

To reduce the hygroscopic character of biodegradable starch-based films, rapeseed oil was incorporated by lamination (starch-oil-starch 3-layers technique). The lipid lamination followed by starch solution casting step induced an emulsion type structure of dried films. Composite films are more opalescent and glossier than fatty free starch films. For all the films, structure is heterogeneous in the cross-section only. Adding fat induced a twice decrease of the tensile strength. Thermal gravimetry analysis did not show differences between films with and without oil. Lipid reduced the moisture absorption particularly at higher RH as well as the surface swelling index, when water droplet contact occurred. Addition of lipids always decreases the contact angle for all liquid tested, except for water. Surface affinity of films for liquids less polar than water increased with rapeseed oil addition. The addition of rapeseed oil significantly reduces water vapour and oxygen permeability.

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1. Introduction

The first films and coatings appeared in China probably in twelfth or thirteenth centuries. Fresh food products were commonly dipped in hot waxes or soy milk skin (Yuba). Nowadays edible films and coatings are produced using a variety of methods and raw-materials: proteins, polysaccharides, lipids and even multi-components as composite structures. Despite of many advantages of each of constituents, carbohydrates and proteins have poor barrier properties. Exceptions are lipids, waxes and resins. Very often lipids are added to film-forming solutions or to films (Baldwin, Hagenmaier, & Bai, 2012). Hydrated food products such as meat, cheeses, sweets, processed foods, fresh or lightly processed fruits and vegetables can lose their organoleptic qualities due to the changes in their water content. To reduce moisture loss, to better control desiccation or to prevent loss of flavour, fatty acids and alcohols, acetylated glycerides, waxes, lacs and cocoa-based compounds and their derivatives, oils have been envisaged as barrier coatings or edible films (Jiménez, Fabra, Talens, & Chiralt, 2012; Parker & Ring, 2000; Savadkar & Mhaske, 2012).

Fats in films might exist as a monolayer (pure fat films) or they can be added to other hydrocolloids and they can create structures in the form of emulsion, dispersion or bi/multilayers (Debeaufort, Quezada-Gallo, & Voilley, 1998). Literature reviews on use of lipids, waxes (such as beeswax, carnauba, candelilla) or resins show that they are often used to coat fresh fruits and vegetables such as oranges, lemons, apples, bananas, plums, mushrooms (Baldwin et al., 2012; Embuscado & Huber, 2009; Jiménez et al., 2012; Rojas-Graü, Soliva-Fortuny, & Martín-Belloso, 2009; Jiménez, Fabra, Talens, & Chiralt, 2013a, 2013b; Jiménez, Sánchez-González, Desobry, Chiralt, & Arab Tehrani, 2013; Linke & Geyer, 2013; Schmidt, Porto, & Laurindo, 2013). Addition of lipids is one of two main ways (the second is cross-linking agents) to reduce the water vapour permeability. Water affinity often controls the water vapour permeability and is related to the hydrophilicity of the film (Wang et al., 2014). Fatty coatings not only retard moisture loss, but also improve appearance by imparting shine to the surface, provide a carrier for fungicides or growth regulators, create a barrier for gas (oxygen, carbon dioxide, ethylene, ...) exchange (Baldwin et al., 2012; Guilbert, 1986; Linke & Geyer, 2013). Indeed, Hall and Sorenson (2006) observed that lipid coatings preserve internal quality of fresh citrus, serve to prevent decay, improve appearance, control weight and carry postharvest fungicides. Rattanapnoe, Plotto, and Baldwin (2007) displayed that

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the colours of lychee was preserved on longer time when fruits were coated by films based on chitosan. Rhim and Shellhammer (2005) applied edible coatings and films based on starch and sunflower oil or stearic acid on fresh-cut products. Chiumarelli and Hubinger (2012) reported that cassava starch films with carnauba wax and stearic acid strongly influenced barrier and mechanical properties and then respiration and preservation of fresh cut apples, favouring their shelf-life.

Finally, from the literature, it seemed that the association of starch and vegetable oil could be a potential challenge for developing edible films for food preservation. Indeed, even if many lipids such as oils, solid fats, and waxes have been tested, very few oils have been used in multilayer films or coatings. Usually they are mixed with hydrocolloids as emulsions. Among the oils, Morillon, Debeaufort, Blond, Capelle, and Voilley (2002) stated that the origin of the oil does not influence significantly the properties of the emulsion-based films. However, very few data are available on the efficiency of oils used in multilayer edible barriers. So the choice of the rapeseed oil is more related to its favourable balance between monosaturated and saturated acids (the lowest among commercial vegetable oils), and its contain of fatty acids (Kairytė & Vėjelis, 2015). Then, rapeseed was chosen as the lipid layer intercalated between two starch layers in this work.

So, the objective of this work is to study the functional properties of composite edible films obtained by laminations of wheat starch solution, rapeseed oil and again wheat starch solution as a 3-layer process, which is original compared to emulsion based edible films. Two concentrations of starch in the film-forming solution were studied, and monolayer starch films were used as controls. Then, structural and physical–chemical characterisation of the films has been conducted.

2. Materials and methods

2.1. Materials

Wheat starch was supplied by Hortimex (Konin, Poland), rapeseed oil from Bouton d'Or (Tourcoing, France) anhydrous glycerol (99.9% of purity) from Sigma–Aldrich (Germany) and ten saturated salt solutions were used for fixing the relative humidity (RH) from ~3 to 93% (from Prolabo, Fontenay-sous-Bois, France).

2.2. Preparation of starch-oil-starch laminate edible films

Wheat starch film-forming solutions were prepared by dissolving 3 g or 5 g of wheat starch powder in 100 mL distilled water. The solutions were heated in a water bath at 85 °C for 30 min under a 700 rpm stirring to obtain complete solubilisation and gelatinization of starch. Film-forming solutions were cooled down to 40 °C. Then the plasticizer was added at a weight ratio of 0.5:1 glycerol:starch. Some preliminary trials have been done, using various content of glycerol. Films with lower glycerol content (i.e. 10–40% of glycerol) were brittle and fractured. Films with higher than 50% of glycerol content had tendency to blooming and to be sticky. Thus, 50% (w/w) of plasticizer contain was considered as the best compromise to get elastic, not breaking, colourless materials. A fixed volume of film-forming solution (30 mL) was poured into a Petri dish ($\varnothing = 13.8 \text{ cm}^2$). Then the dry matter per surface unit, and thus the final thickness varied according the film-forming solution composition. Films were dried at 25 °C and 30% relative humidity (RH) for 48 h in a climatic chamber (KBF 240, binder, Germany). After drying, a layer of rapeseed oil (3 g) was deposited by brushing. The layer of rapeseed oil was spread and kept in previous aforementioned conditions for 24 h. After this time, a third layer of the starch film-forming solution was cast and dried for

48 h in same conditions. Dry films were peeled off and stored at $53 \pm 1\%$ RH and 25 ± 1 °C in desiccators containing saturated magnesium chloride for at least 7 days prior to testing.

So films were coded as 3S, 3L and 5S, 5L, respectively according their starch content in film forming solution (3 or 5 g of starch in 100 mL of water) and oil layer addition (L) or not (S). To summarise, the dry compositions of films: 3S and 5S have a 1:0.5 starch:glycerol ratio, 3L has a 1:0.5:1 starch:glycerol:oil ratio and the 5L has a 1:0.5:0.6 starch:glycerol:oil ratio.

2.3. Colour

Colour of films was determined using a colorimeter (Minolta, Model CR-300, Japan) using the CIE LAB colour parameters: L , from black (0) to white (100); a , from green (–) to red (+); and b , from blue (–) to yellow (+) (Gennadios, Weller, Hanna, & Froning, 1996). Colour of films was expressed as the total colour difference (ΔE) according to the following equation (Sobral, dos Santos, & Garcia, 2005):

$$\Delta E = \sqrt{(L^* - L)^2 + (a^* - a)^2 + (b^* - b)^2} \quad (1)$$

where $L^* = 96.74$, $a^* = 0.09$, $b^* = 2.20$ are the colour parameter of a white standard plate used as the film background.

2.4. Structure characterizations

2.4.1. Film microstructure

Film microstructure was observed using an environmental scanning electron microscope (ESEM, Philips XL 30 ESEM, Japan). A $0.5 \times 1.0 \text{ cm}$ film was fixed on the support using double side adhesive, at an angle of 90° to the surface which allowed observation of the cross-section of the film. All the film samples were cut with a new razor blade to prevent as much as possible morphological damage. Films were focused up to 15,000 \times , and magnifications ranging from 800 \times to 8000 \times where selected, with an intensity of 8 kV and absolute pressure of 230 Pa in presence of water (RH ~ 30% at 5 °C). Any special preparation as palladium or gold coating was necessary for ESEM observations.

2.4.2. Film thickness

Film thickness was measured with a PosiTector 6000 (DeFelsko, USA) digital micrometre to the nearest 1 μm in 0–100 μm range. Prior to film thickness measurements the electronic gauge was calibrated at 74 and 139 μm using standards. Thickness of each film was measured in five places, one in the centre part of the film and four around its perimeter, and an average value was used in the calculations. Thickness was measured in 5 places on each film peeled from the Petri dishes. At least 10 replications of each formulation have been made.

2.4.3. Mechanical properties

Tensile strength and elongation at break of the films ($10 \times 2.5 \text{ cm}$) were measured ten times using a TA-XT2i Texture Analyser (Stable Microsystems, United Kingdom) according to the ASTM D882-95 method (ASTM, 1995). Self-tightening roller grips were used to perform tensile tests. The initial distance between the grips and the initial velocity were adjusted to 50 mm and 1 mm s^{-1} . Measurements were performed at least 15 times on each film recipe. Mechanical properties were calculated using the average thickness of each film sample.

2.4.4. Thermal stability

The thermal stability of film was assessed by Thermogravimetry. Dried films were scanned using a thermogravimetric analyser (TGA-7, Perkin Elmer, Norwalk, CT, USA) from 40 to

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