



# Effect of starch type on the physico-chemical properties of edible films



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## ABSTRACT

Food preservation is mostly related to packaging in oil-based plastics, inducing environmental problems, but this drawback could be limited by using edible/biodegradable films and coatings. Physical and chemical properties were assessed and reflect the role of the starch type (wheat, corn or potato) and thus that of the amylose/amylopectin ratio, which influences thickness, colour, moisture, wettability, thermal, surface and mechanical properties. Higher amylose content in films induces higher moisture sensitivity, and thus affects the mechanical and barrier properties. Films made from potato starch constitute a greater barrier for oxygen and water vapour though they have weaker mechanical properties than wheat and corn starch films. Starch species with higher amylose content have lower wettability properties, and better mechanical resistance, which strongly depends on the water content due to the hydrophilic nature of starch films, so they could be used for products with higher water activity, such as cheese, fruits and vegetables. It especially concerns wheat starch systems, and the contact angle indicates less hydrophilic surfaces (above 90°) than those of corn and potato starch films (below 90°). The starch origin influences optical properties and thickness: with more amylose, films are opalescent and thicker; with less, they are transparent and thinner.

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## 1. Introduction

The most suitable, abundant, renewable and low-cost material for producing edible coatings based on biopolymers is starch [1,2]. Due to this fact, there is a growing need to understand the interactions between the film structure, water molecules and other constituents used for edible film making. Starch is a very well-known carbohydrate polymer, and plants from which it is obtained grow in nearly all the temperate zones [3]. Corn, wheat, potato and rice take the world leading positions: 84%, 7%, 4% and 1% respectively. Tapioca, yam, oat, sweet potato, barley, rye, taro, millet, amaranth grain, quinoa, fava, lentils, mung beans, peas, chickpeas, palms, chestnuts, acorns, arrowroot, arracacha, bananas, breadfruit, buckwheat, canna, colocasia, katakuri, kudzu, malanga, oca, Polynesian arrowroot, sago, sorghum, and water chestnuts are the other starch sources that could be envisaged for such applications

[4–7]. Amylose is a sparsely branched carbohydrate mainly based on  $\alpha(1-4)$  bonds with a molecular weight of  $10^5$  to  $10^6$  anhydroglucose units. The number of macromolecular configurations based on  $\alpha(1-6)$  links is directly proportional to the amylose molecular weight [8]. Amylopectin is a highly multiple-branched polymer with a high-molecular weight of  $10^7$  to  $10^9$  anhydroglucose units. Depending on the botanical resource starch granules vary in size from about 2–150  $\mu\text{m}$ , the amylose content from 0% to about 75%, but typical is 20–25% (w/w) [9]. The waxy starch contains low or no amylose, whereas high-amylose starch contains more than 50% of the linear polymer. Chemical, physical and functional properties of edible films and coatings depend on the amylose and amylopectin ratio [10,11]. Palviainen et al. [12] described better mechanical and gas barrier properties of starch with high amylose content such as from pea. Joshi et al. [13] noted that compared to corn and potato starches, lentil starch (30% amylose) possesses a strong gel-forming tendency at a relatively low concentration. Whatever the botanical origin, starch exhibits several disadvantages such as strong hydrophilic character (water sensitivity), which make it unsatisfactory for some applications.

The objective of this study was to investigate microstructural, optical, mechanical, barrier and thermal properties of 3 types of

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films made from corn, potato and wheat starch and containing 33% (starch dry basis) glycerol. Additional goals of this study were to:

- 1 determine the difference in amylose content for wheat, potato and corn starches. Although the difference is quite small it has a significant influence on some physical, chemical and functional properties.
- 2 determine the dependences and relations of amylose/amylopectin fractions on selected chemical, physical and functional properties of starch films.

## 2. Materials and methods

### 2.1. Materials

Wheat (25% amylose), corn (27% amylose) and potato (20% amylose) starches were supplied by Hortimex (Konin, Poland), anhydrous glycerol (99.9% of purity) from Sigma-Aldrich (Germany) and nine saturated salt solutions (all Prolabo, Fontenay-sous-Bois, France) were used for fixing the relative humidities (RH) at 25 °C: lithium chloride (11%), potassium acetate (22%), magnesium chloride (33%), potassium carbonate (43%), magnesium nitrate (53%), sodium nitrite (65%), sodium chloride (75%), ammonium sulphate (81%) and ammonium dihydrogen phosphate (93%).

### 2.2. Preparation of starch films

Wheat or corn or potato starch film-forming solutions were prepared by dissolving 5 g of starch powder (wet basis) in 100 mL of distilled water. The solutions were heated in a water bath at 85 °C for 30 min under 700 rpm stirring to obtain complete solubilisation and gelatinization of starch. Then, film-forming solutions were cooled down to 40 °C. The plasticizer was added at a weight ratio of 0.3:1 glycerol:starch under stirring at 150 rpm. The concentration of plasticiser was chosen according to a preliminary experiment, i.e., 30% to the starch weight is the minimum concentration required to obtain ductile and not brittle films. Biomaterials with addition of glycerol more than 50% to starch weight become soggy and white (overplasticized). A defined volume of film-forming solution was poured into a Petri dish to obtain a constant film thickness whatever the composition. Films were dried at 25 °C and 30% relative humidity (RH) for 48 h in a climatic chamber (KBF 240, Binder, Germany). Dry films were peeled off and stored at 53 ± 1% RH and 25 ± 1 °C in desiccators containing saturated magnesium chloride for at least 7 days prior to any testing.

### 2.3. Film characterisation

#### 2.3.1. Film thickness

Film thickness was measured with a PosiTector 6000 (DeFelsko, USA) digital micrometer to the nearest 1 µm in the 0–100 µm range and to the nearest 5 µm in the 100–1000 µm range. Prior to film thickness measurements the electronic gauge was calibrated at 74 and 139 µm using standards to cover the range of film sample thicknesses. Thickness of each film was measured just after peeling from the Petri dish in room conditions (35–45% RH and ~25 °C). Measurements were done in five places (one in the centre of the film and four around its perimeter) in 10 replications at least. For calculations the mean value was used.

#### 2.3.2. Film observations: SEM and colour

Film microstructure was observed using an environmental scanning electron microscope (ESEM, Philips XL 30 ESEM, Japan). A 0.5 × 1.0 cm film was fixed on the support using double sided 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 any morphological damage. Films were focused up to ×15 000, and magnifications ranging from ×800 to ×8000 were selected, with an intensity of 8 kV and absolute pressure of 230 Pa in the presence of water (RH ~30% at 5 °C). No special preparation such as palladium or gold coating was necessary for ESEM observations.

Colour of films (20 measurements were taken) 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 (+) [14]. Colour of films was expressed as the total colour difference ( $\Delta E$ ) from a standard white colour ( $L^* = 96.74$ ,  $a^* = 0.09$ ,  $b^* = 2.20$ ) and used as the film background, according to the following equation [15]:

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

#### 2.3.3. Thermo-gravimetric analysis (TGA)

The films were scanned using a thermogravimetric analyser (TGA-7, Perkin Elmer, Norwalk, CT, USA) from 40 to 800 °C at a rate of 10 °C/min. Nitrogen was used as the purge gas at a flow rate of 20 mL/min. Measurements were done in 3 replications. Before the tests, the films were equilibrated at 53% of RH for at least one week.

#### 2.3.4. Contact angle determination

The contact angle ( $\theta$ ) was measured at the triple contact point of liquid/solid/air phases. It describes the relationship between the surface tension energies of three phases according to the Young equation [16].

To measure the contact angle the sessile method drop was used: a droplet (about 1.5 µL) of a test liquid was placed on a horizontal film surface. Measurements were done using a DGD-DX goniometer equipped with the DIGIDROP image analysis software (GBX, Romans-sur-Isere, France), according to the methodology of Karbowiak et al. [17]. The contact angle was measured on both sides of the drop and averaged. The contact angle and drop volume measurements were carried out over 120 s. The effect of evaporation was assessed on an aluminium foil considered as an impermeable reference surface and subtracted from the sample. Then, the rate of evaporation was considered in the study of the kinetics of wetting and absorption. Measurements for all samples were made on the side of the films exposed to air during drying in order to prevent the support (Petri dish) effect. Measurements were made in a minimum of 10 replications. Before analysis, films were equilibrated in 53% relative humidity, for at least 7 days, and were kept there until measurements. During the tests biomaterials were characterised at room conditions (25 °C and 35% of RH).

#### 2.3.5. Film surface tension and critical surface tension determination

The surface tension of the liquid tested ( $\gamma_L$ ) was measured by the sessile drop method and Laplace-Young approximation [18,19]. The estimation of the critical surface tension ( $\gamma_C$ ) of the starch-based films was obtained from the Zisman plot [20] as previously used by Basiak et al. [21]. For determination of the surface energy and its components the following liquids with known polar and dispersive constituents were used: cyclopentanol, diiodomethane, ethylene glycol, methyl benzoate, *n*-octane, polyethylene glycol, tetradecane, water and 1-bromonaphthalene.

The surface tension of films ( $\gamma_S$ ) and their dispersive ( $\gamma_S^D$ ) and polar ( $\gamma_S^P$ ) components were calculated using the Owens-Wendt method [22] according to equations (2) and (3):

$$\gamma_S = \gamma_S^P + \gamma_S^D \quad (2)$$

$$\gamma_L = (1 + \cos \theta) = 2 \left( \sqrt{\gamma_L^P \gamma_S^P} + \sqrt{\gamma_L^D \gamma_S^D} \right) \quad (3)$$

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