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Humidity stability of tapioca starch-pullulan composite films

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ABSTRACT

Pure and composite films based on tapioca starch and pullulan were investigated in their stability against dry and humid storages (RH 23 and 85%). The films were prepared by casting aqueous dispersions of pullulan and starch mixtures at different ratios: 5% starch and 0–20% pullulan. For the pure pullulan film, more than 10% solids were required to produce a homogeneous film, whereas a rigid and homogenous film could be produced with starch even at 5% concentration. The pullulan film (20% solids) was highly susceptible to the exposure to the humid condition (RH 85%), whereas the composite films containing starch (5%) and pullulan (2–10%) exhibited improved stability with reduced moisture absorption and less deformation during the ambient storage at 85% RH for 2 weeks. As the starch incorporation increased, the films became more rigid showing increases in the tensile strength but decreases in the elongation at break. By using a proper composite ratio (5% starch, and 2 or 5% pullulan), the film could be optimally prepared to exhibit both mechanical strength and storage stability against humidity. However, the starch tended to decrease the water solubility of the composite films.

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1. Introduction

Pullulan is a water-soluble polysaccharide produced by fungus strains, Aureobasidium pullulans and Pullularia pullulans (Shibata, Asahina, Teramoto, & Yosomiya, 2001) It consists mainly of maltotriose units polymerized in a linear fashion via α -1, 6-glucosidic linkages which endows pullulan with structural flexibility and good water solubility (Leathers, 2003). It may also be used as a carrier for bioactive compounds and a protective packaging for food and pharmaceutical components and products (Tong, Xiao, & Lim, 2008). Pullulan may also be used as a partial replacement for starch in processed foods such as baked or pasted products, and various oral care products (Leathers, 2003). Pullulan is an ideal material for the preparation of edible films because it is non-toxic, non-mutagenic, odorless, and tasteless (Kimoto, Shibuya, & Shiobara, 1997; Leathers, 2003). Pullulan-based films are characterized with excellent mechanical properties and a low permeability to oil and oxygen (Leathers, 2003; Tong et al., 2008). The hydrophilic nature of pullulan, however, often causes a substantial decrease in mechanical strength of the film when exposed to elevated relative humidity (RH) (Shibata, Nozawa, Teramoto, &

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http://dx.doi.org/10.1016/j.foodhyd.2014.04.008 0268-005X/© 2014 Elsevier Ltd. All rights reserved. Yosomiya, 2002; Tong et al., 2008). Therefore, the edible films prepared with pullulan, which are commercially available in markets, are highly sensitive to humidity changes, becoming brittle when exposed to dry environments and sticky under humid conditions (Kawahara et al., 2003; Shibata et al., 2002; Wiley et al., 1993). Furthermore, the relatively high cost of pullulan itself compared to other biopolymers such as starch and cellulose derivatives limits its utilization for the edible films (Tong et al., 2008; Xiao, Lim, & Tong, 2012).

Starch is one of the biopolymers that could be utilized for film preparation, often in composition with other polymers. It can be used as a raw material for films at a much lower cost than pullulan. Starchbased films were reported to exhibit physical characteristics similar to those of the films prepared of synthetic polymers in transparency and permeability of various gas components (Krochta, Baldwin, & Nisperos - Carriedo, 1994; Xu, Kim, Hanna, & Nag, 2005). Among the commercial starches isolated from different origins, tapioca starch has been widely used in foods and other industrial commodities because it has superior properties to cereal starches such as corn and wheat starches. In native form, tapioca starch may produce coatings and films with good transparency and flexibility without a special plasticizer (Bergo et al., 2008; Flores, Famá, Rojas, Goyanes, & Gerschenson, 2007; Vicentini, Dupuy, Leitzelman, Cereda, & Sobral, 2005). However, the inferior resistance to dry and humid conditions of the starch-based films limits the utilization of the films in food and pharmaceutical industries (Wu & Zhang, 2001).









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It has been reported that the composites of two or more biopolymers may enhance the physical properties and water resistance of the films. Composite films prepared with starch and chitosan displayed an improvement in mechanical strength and decrease in water vapor transmission. It was also reported that pullulan–alginate–carboxymethylcellulose composite films exhibited a similar improvement in such physical properties (Kristo & Biliaderis, 2007; Tong et al., 2008; Vásconez, Flores, Campos, Alvarado, & Gerschenson, 2009; Xu et al., 2005). Modified starch blended with soy protein could be used for film preparation, and the composite films were reported to have enhanced elongation compared to that of the pure starch film (Galus, Mathieu, Lenart, & Debeaufort, 2012).

In the present study, tapioca starch and pullulan blends were used for the preparation of edible films, and the mechanical strength and stability of the composite films were investigated in their stability against dry and humid storages.

2. Materials and methods

2.1. Materials

Tapioca starch was provided by Samyang Genex Company (Seoul, South Korea) and pullulan (MW 200,000) was obtained from Hayashibara Biochemical Laboratory (Okayama, Japan). The CH₃COOK, Mg(NO₃)₂, and KCl were of analytical grade.

2.2. Pasting viscosity and film preparation

The pasting viscosity of film-forming dispersions was determined using the Rapid Visco-Analyzer (RVA; Newport Scientific, Warriewood, Australia). Tapioca starch content remained constant at 5% (dry solids) whereas pullulan content in the dispersion varied from 0 to 10% (dry solids in total dispersion). For comparison, a pullulan film (20% solids), similar to the commercial edible films, was also prepared. Each mixture of starch and pullulan was transferred to an aluminum container for RVA analysis, and then distilled water was added into the mixture until the total weight reached to 30 g. The dispersion was held at 50 °C for 1 min, heated to 95 °C at a rate of 12.2 °C/min, maintained at 95 °C for 8 min, cooled to 50 °C at 12.2 °C/min, and then held at 50 °C for 1 min under continuous shear at 160 rpm.

For the film preparation, the aqueous dispersions of starchpullulan mixtures were pasted according to the procedure described for RVA analysis without the final cooling and holding steps. The hot paste was centrifuged for 2 min at $840 \times g$ to remove air bubbles, and then was casted to films by a film applicator (COAD 411, Uiwang, South Korea). The films prepared were then dried at 50 °C and RH 50% for 2 h prior to analyses.

2.3. Thickness and transparency

Thickness of the dried films was measured by a micrometer caliper (Mitutoyo Manufacturing Co. Ltd., Kawasaki, Japan). At least 3 different positions on the film were used to take measurements for each film sample.

Transparency of the films was determined according to the method described by Han and Floros (1997). The film was cut into a rectangular shape (4 mm \times 1 cm) and placed inside a spectrophotometer cell, and then the percent light transmittance of the film was analyzed at 600 nm by using a UV spectrophotometer (model UV-2100, Pgeneral Instrument Co. Ltd., Beijing, China). The transparency was calculated by the following equation: Transparency = (log%*T*)/*b*, where %*T* was the light transmittance at 600 nm and *b* was the thickness of film (mm).

2.4. Moisture content

Square specimens (20×20 mm) of the films prepared with different composites of starch and pullulan were stored in the desiccators containing the saturated salt solutions: CH₃COOK, Mg(NO₃)₂, and KCl for constant RH of 23, 53, and 85%, respectively (Bertuzzi, Castro Vidaurre, Armada, & Gottifredi, 2007). After 6 days of storage at 25 °C, the film samples were taken, and the moisture content of the films was determined by drying at 105 °C in a convection oven for 3 h.

2.5. Mechanical strength

Tensile properties of the films were determined by using a TA-XT2i texture analyzer (Stable Micro System, Surrey, UK) in accordance with the ASTM D882-02 guidelines (ASTM, 2002) with minor modifications. All of the tested films were cut into a strip (30×50 mm) and the strips were equilibrated by storing at 25 °C under various RH conditions (23, 53, and 85%) for 6 days prior to testing. The tensile strength and elongation at break were measured with the strips during the extension up to breakage at 1 mm/s with an initial distance of 35 mm. Average thickness of the films was measured after moisture equilibration as described previously.

2.6. Water solubility

The films were cut into square specimens (20×20 mm) and the specimens were equilibrated by storing at 25 °C under 53% RH until reached constant weight. The specimens were placed in distilled water (50 mL) and then the dispersion was magnetically stirred at 37 °C (300 rpm). At regular intervals (15, 30, and 60 s), aliquots of the solution (1 mL) were collected, and the total carbohydrate content in the solution was determined using the phenol–sulfuric acid method (DuBois, Gilles, Hamilton, Rebers, & Smith, 1956). The solubility of films was expressed as percent ratio of the soluble carbohydrate to the initial solid weight of the film.

2.7. Experimental design and statistical analysis

All treatments consisted of two full treatment replications, and individual analyses for each replicate were conducted in triplicate. Duncan's test was used to determine differences among experimental mean values (p < 0.05). Pearson's correlation analysis was used to examine relationships among experimental factors. All statistical analyses were conducted using SAS version 9.1 for Windows (SAS Institute, Cary, NC, USA).

3. Results and discussion

3.1. Pasting viscosity of starch-pullulan mixtures

Pasting viscosity of a film-forming dispersion is one of the important factors determining the morphology and physical properties of the film (Debeaufort, Quezada-Gallo, & Voilley, 1998; Gennadios, Weller, & Testin, 1993). Fig. 1 shows the pasting viscosity of the film-forming dispersions containing different starch–pullulan mixtures. The dispersions containing pure pullulan formed a viscous paste when the pullulan content was 20%, and did not form a viscous paste when the concentration was 10% or less. It was because pullulan typically exhibits a much lower viscosity than other hydrocolloids at the same concentration (Leathers, 2003). The pullulan paste showed a substantial thinning during the mechanical stirring at the hot stage (95 °C), which was typical for the aqueous pastes of most hydrocolloids. On the other hand, tapioca

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