



Oxidation of potato starch with different sodium hypochlorite concentrations and its effect on biodegradable films



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ARTICLE INFO

Article history:

Received 17 June 2014

Received in revised form

4 October 2014

Accepted 23 October 2014

Available online 31 October 2014

Keywords:

Carbonyl

Carboxyl

Films

Oxidation

Potato starch

ABSTRACT

The objective of this study was to evaluate the effects of oxidation with sodium hypochlorite at different concentrations of active chlorine (0.5, 1.0 and 1.5 g/100 g) on the physicochemical, morphological, pasting, thermal and gel texture properties of potato starch. The native and oxidized starches were used in different concentrations (3.0, 4.0 and 5.0 g starch/100 mL) for the preparation of biodegradable films using glycerol as plasticizer. The films were evaluated for their mechanical properties, color, water solubility and permeability of water vapor. The degree of oxidation potato starches was more intense as there was an increased concentration of active chlorine in the reaction. Films made with oxidized starch with the highest level of active chlorine had lower tensile strength when compared to native starch films. Also, these films had a lower water solubility and water vapor permeability as compared to the native starch films.

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1. Introduction

Starch is a renewable raw material, abundant and available in all the world, and shows properties that are modifiable from the physical (Zavareze & Dias, 2011), chemical (Kuakpetoon & Wang, 2001; Menzel et al., 2013) and enzymatic (LeCorre, Vahanian, Dufresne, & Bras, 2012; Rajan & Abraham, 2006) or combined (Spier, Zavareze, Silva, Elias, & Dias, 2013) ways, which can result in resistant films and coatings (Cyras, Zenklusen, & Vazquez, 2006; Mali, Grossmann, Garcia, Martino, & Zaritzky, 2004; Zavareze et al., 2012). Some studies about starch films' functional properties characterization have been developed, with the result that the biggest technological problems and application are high water vapor permeability and low mechanical resistance. One way to improve these properties of starch films is to modify the chemical structure of the starch molecules (Hu, Chen, & Gao, 2009).

Although the main application for oxidized starch is in the paper and textile industries, its use in other areas is increasing due to its low viscosity at a high solid concentration, high paste transparency (Wang & Wang, 2003), high stability and good film forming

capacity (Sangseethong, Lertphanich, & Sriroth, 2009). The starch oxidation allows the formation of carbonyl and carboxyl groups in glucan chain by a hydroxyl groups substitution (Wang & Wang, 2003). Some different oxidizing agents can be used to oxidize starches, such as sodium hypochlorite and hydrogen peroxide (Dias et al., 2011; Hu et al., 2009; Klein et al., 2014; Kuakpetoon & Wang, 2001; Wang & Wang, 2003). Following oxidation, the relatively bulky carboxyl (COOH) and carbonyl (CO) groups are introduced with a partial depolymerization of the starch chains (Chan, Bhat, & Karim, 2009) resulting in a low retrogradation of oxidized starch paste, whereas carbonyls play a minor role in the prevention of the retrogradation of starches (Sandhu, Kaur, Singh, & Lim, 2008). This conformation can increase the free space between glucan molecules and it is an important property for the plasticizer interaction during the starch filming formation.

Numerous research about starch oxidation was done with different raw materials, such as corn, potato, cassava, and beans (Spier et al., 2013; Vanier et al., 2012; Wang & Wang, 2003; Zavareze et al., 2012). However, a few studies were done using oxidized starches to produce biodegradable films. The most recent research is about potato and banana oxidized starches (Hu et al., 2009; Zamudio-Flores, Vargas-Torres, Pérez-González, Bosquez-Molina, & Bello-Pérez, 2006).

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Properties of biodegradable starch films are dependent on starch raw material, starch concentration on polymeric suspension, the type and modification conditions, the type and concentration of the plasticizer used, as well as drying conditions. [Zavareze et al. \(2012\)](#) made potato starch films using oxidized and HMT (heat–moisture treatment) starches; however, they used only one modification degree to produce the films. Therefore, the aim of this study was to assess the effect of the different sodium hypochlorite concentration upon the starch physicochemical, morphological, thermal, paste and gel texture properties, the film forming capacity, water solubility, mechanical properties, and the water vapor permeation of the films.

2. Material and methods

2.1. Material

Potatoes from the Baronesa (*Solanum tuberosum* L.) cultivar were used. Potato starch was isolated according to the method described by [Liu, Weber, Currie, and Yada \(2003\)](#). Potato samples were dehulled and soaked in 0.1 g/100 g sodium bisulphite at a 1:7 (g:mL) ratio for 2 h after the dispersion was ground in a blender (Britania, Brazil) for 5 min, passed through a 63 µm screen and decanted. The starch was washed a minimum of three times with distilled water and dried at 40 °C until the moisture content of the samples reached approximately 9 g/100 g.

2.2. Starch oxidation

Starch oxidation was performed according to the method described by [Dias et al. \(2011\)](#), with some modifications. A starch sample (200 g dry basis (d.b.)) was suspended in 500 mL of distilled water in a glass reactor of 1 L, heated at 40 °C and subjected to a sodium hypochlorite treatment. The pH of the starch slurry was adjusted to 7.0 with 0.5 mol equi/L NaOH and 0.5 mol equi/L HCl. After the addition of sodium hypochlorite, the pH value of the slurry was maintained at 7.0 with 0.5 mol equi/L NaOH for an additional 60 min. The slurry was then filtered by suction with a Buchner filter funnel, washed with a twofold volume of distilled water and dried in a convection oven at 40 °C for 16 h until there was approximately 9 g/100 g moisture content. The same procedure was applied for different active chlorine concentrations (0.5, 1.0 and 1.5 g/100 g).

2.3. Amylose content

The amylose content was determined by the colorimetric method with iodine according to the method of [McGrance, Cornell, and Rix \(1998\)](#), with the modifications suggested by [Hoover and Ratnayake \(2001\)](#). Starch (20 mg, dry basis) was dissolved in 8 mL of dimethylsulfoxide (90 g/100 mL) in 10 mL screw–cap reaction vials. The contents of the vials were vigorously mixed for 20 min and then heated in a water bath (with intermittent shaking) at 85 °C for 15 min. The vials were then cooled to ambient temperature, and the contents diluted with water to 25 mL in a volumetric flask. The diluted solution (1 mL) was mixed with water (40 mL) and 5 mL I₂/KI solution (0.0025 M I₂ and 0.0065 M KI) and then adjusted to a final volume of 50 mL. The contents were allowed to stand for 15 min at ambient temperature, before absorbance measurements at 600 nm.

2.4. Carbonyl and carboxyl contents

The carbonyl content was determined according to the titrimetric method adapted by [Vanier et al. \(2012\)](#). A starch sample (2 g)

was added to 100 mL of distilled water in a 500-mL flask. The suspension was gelatinized in a boiling water bath for 20 min, cooled to 40 °C, and adjusted to a pH value of 3.2 with 0.1 mol equi/L HCl. A hydroxylamine reagent (15 mL) was then added to the mixture. The flask was stoppered and placed in a 40 °C water bath for 4 h with slow stirring. The excess hydroxylamine was determined by rapidly titrating the reaction mixture to a pH value of 3.2 with standardized 0.1 mol equi/L HCl. A blank determination with only the hydroxylamine reagent was performed in the same manner. The hydroxylamine reagent was prepared by first dissolving 25 g of hydroxylamine hydrochloride in 100 mL of 0.5 mol equi/L NaOH, before the final volume was adjusted to 500 mL with distilled water. The carbonyl content was expressed as the quantity of carbonyl groups per 100 glucose units (CO/100 GU), as calculated by Equation (1).

$$\text{CO}/100\text{GU} = \frac{(V_b - V_s) \times M \times 0.028 \times 100}{W} \quad (1)$$

where V_b is the volume of HCl used for the blank (mL), V_s is the volume of HCl required for the sample (mL), M is the molarity of HCl and W is the sample weight (dry basis).

The carboxyl content of the oxidized starch was determined according to the modified procedure of [Chattopadhyay, Singhal, and Kulkarni \(1997\)](#). Approximately 2 g of a starch sample was mixed with 25 mL of 0.1 mol equi/L HCl, and the slurry was stirred occasionally for 30 min with a magnetic stirrer. The slurry was then vacuum filtered through a 150 mL medium porosity fritted glass funnel and washed with 400 mL of distilled water. The starch cake was then carefully transferred into a 500 mL beaker, and the volume was adjusted to 300 mL with distilled water. The starch slurry was heated in a boiling water bath with continuous stirring for 15 min to ensure complete gelatinization. The hot starch dispersion was then adjusted to 450 mL with distilled water and titrated to a pH value of 8.3 with standardized 0.01 mol equi/L NaOH. A blank test was performed with unmodified starch. The carboxyl content was expressed as the quantity of carboxyl groups per 100 glucose units (COOH/100 GU), as calculated by Equation (2).

$$\text{COOH}/100\text{GU} = \frac{(V_b - V_s) \times M \times 0.045 \times 100}{W} \quad (2)$$

where V_s is the volume of NaOH required for the sample (mL), V_b is the volume of NaOH used to test the blank (mL), M is the molarity of NaOH and W is the sample weight (dry basis).

2.5. Scanning electron microscopy (SEM)

The morphology of the starch granules was examined using a Shimadzu SSX-550 scanning electron microscope. The starch samples were initially suspended in acetone at 1 g/100 mL. A small quantity of each sample was spread directly on the surface of the stub and dried in an oven at 32 °C for 24 h. Subsequently, all of the samples were coated with gold and examined using a scanning electron microscopy under an acceleration voltage of 15 kV at a magnification of 200×.

2.6. Pasting properties

The pasting properties of the starch samples were determined using a Rapid Visco Analyser (RVA–4, Newport Scientific, Australia) with a Standard Analysis 1 profile. The viscosity was expressed in rapid visco units (RVU). The starch (2.0 g of 14 g/100 g wet basis) was weighed directly in the RVA (Rapid Visco Analyser) canister, and 25 mL of distilled water was then added to the canister. The

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