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Acetylation of rice starch in an aqueous medium for use in food

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ABSTRACT

The degree of acetylation controls the use of starch acetate, since the FDA (Food and Drug Administration USA) recommends an acetyl groups' percentage below 2.5 g/100g for a food application. The objective of this work was to evaluate the effects of the acetic anhydride concentration, in an aqueous solution, on the degree of acetylation, in physical, thermal, pasting and morphological properties, and enzymatic susceptibility on rice starch acetylated with the acetyl groups' percentage up to 2.5 g/100g. The rice starch was acetylated using different concentrations of acetic anhydride (5 g/100g, 10 g/100g and 20 g/ 100g, starch db). Acetylation increased the peak viscosity, breakdown, final viscosity, retrogradation and pasting temperature and reduced the hardness of the gels, as well as their adhesiveness and gumminess; however this did not affect the morphology and susceptibility to hydrolysis by the α -amylase of the starch. The acetylated starches using 10 and 20 g/100g acetic anhydride showed lower swelling power and solubility compared to native starch.

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1. Introduction

Starch is the major constituent of rice grains and it is an important ingredient in food preparation (Bao, Kong, Xie, & Xu, 2004; Blazek & Gilbert, 2010). The rice starch has been widely used as an ingredient in various food and industrial products such as desserts, bakery products and alternative fats. Also, it has been used as an ingredient for the production of breakfast cereals, hypoallergenic products, and infant formulas (Puchongkavarin, Varavinit, & Bergthaller, 2005), due to the hypoallergenicity of the associated protein. Rice starch with high amylose content has a low glycemic index too (Champagne, 1996).

The starch in its native form has limited applications; therefore, it does not always have the desired properties for certain types of processing; but when modified, it increases its range of use. The chemically modified starch by acetylation has been widely studied (Bello-Pérez, Agama-Acevedo, Zamudio-Flores, Mendez-Montealvo, & Rodriguez-Ambriz, 2010; Chen, Li, Li, & Guo, 2007; Gunaratne & Corke, 2007; Huang, Zhang, Chen, & Li, 2010; Luo & Shi, 2012; Mbougueng et al., 2012; Singh, Chawla, & Singh, 2004; Singh & Nath, 2012). The acetylation may be performed to

improve the physical, chemical and functional properties of starch (Xu, Miladinov, & Hanna, 2004). In the acetylation process, the hydroxyl groups of the glucose monomers are converted to the groups CH₃COO- (Graaf, Broekroelofs, Janssen, & Beenackers, 1995); therefore the acetylation is an esterification of hydroxyl groups in the anhydroglucose unit of the starch molecule.

The starch acetate has applications which are regulated by their characteristics, such as the degree of acetylation or the degree of substitution (DS), and the percentage of acetyl groups (% Ac). These characteristics determine the use of starch acetate, for example, for food application; the FDA (Food and Drug Administration USA) recommends a percentage of acetyl groups of less than 2.5 g/100g.

The changes promoted by acetylation depend on the botanical source, the degree of substitution, the proportion of amylose/ amylopectin and the molecular structure of starch. In the acetylation reaction, the number of acetyl groups incorporated in the starch molecule and the efficiency of the reaction depend on the type of reagent, concentration, pH, presence of catalyst, reaction time, botanical origin and the characteristics of the size and structure of the starch granules (Huang, Schols, Jin, Sulmann, & Voragen, 2007; Huber & BeMiller, 2000).

According to Mark and Mehltretter (1972), the starch acetates, in accordance with DS, are classified as low DS (<0.1), medium DS (0.1–1.0) and high DS (>1.0) acetated. There is a commercial interest in starches with a DS of 0.01–0.20 because of their use based







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on properties to film formation, adhesion, thickening, stabilizing and texturizing (Boutboul, Giampaoli, Feigenbaum, & Ducruet, 2002). The acetylated starch with a low DS is commonly obtained by the esterification of native starch with acetic anhydride, in an aqueous medium, and in the presence of an alkaline catalyst.

The applicability of acetylated starches is completely dependent on the degree of substitution. The introduction of acetyl groups interrupts the ordered structure of native starch and interferes with the reassociation of amylose and amylopectin molecules in the gelatinized starch, leading to a decrease in the gelatinization temperature, an increase or decrease in the swelling power, and solubility along with the storage stability.

Previous studies on acetylated rice starch have been reported by Colussi et al. (2014) and Gonzalez and Perez (2002). Colussi et al. (2014) studied the effect of acetylated rice starch varying the amylose content with the different reaction times. Gonzalez and Perez (2002) evaluated the effect of acetylation in the chemical composition, and the physical, chemical, functional and rheological properties of rice starch. However, to our knowledge, there are no studies in literature on the acetylation of rice starch with an acetylation degree allowed by legislation (FDA) for food applications. Therefore, this work aims to produce rice starch acetylated with a percentage of acetyl groups up to 2.5 g/100g to allow for use in foods and evaluate the influence of the acetic anhydride concentration on the pasting, physical, thermal and morphological properties, and enzymatic susceptibility.

2. Materials and methods

2.1. Material

Rice starch of a high amylose (32% amylose), cultivar IRGA 417, was extracted according to the method proposed by Wang and Wang (2004).

2.2. Synthesis of starch acetates in an aqueous medium

The starches were acetylated according to the method proposed by Phillips, Huijum, Duohai, and Harold (1999). A suspension of 50 g of rice starch in 112 mL of distilled water was subjected to shaking at 1500 rpm (RW 20, IKA, Germany) for 60 min at 25 °C. The pH of the suspension was adjusted to 8.0 with an aqueous 3.0 g/ 100g NaOH solution, and a slow addition of 5, 10 and 20 g/100g of acetic anhydride (starch db), respectively, maintaining the pH between 8.0 and 8.4 with a 3.0 g/100g NaOH solution. After the complete addition of acetic anhydride, the reaction was continued for 15 min. The reaction was stopped by adjusting the pH to 4.5 with a 0.5 mol equi/L HCl solution. The final suspension was centrifuged for 3 min at 1000 × g and subjected to successive washings with 95 mL/L ethyl alcohol. The starch was dried in an oven with air circulation at 40 °C where the humidity reached about 10 g/100g.

2.3. Determination of the acetyl percentage (Ac%) and degree of substitution (DS)

The percentage of acetyl groups (Ac%) and the degree of substitution (DS) of the acetylated starches were determined by the titration method described by Wurzburg (1964). Acetylated starch (1 g) was mixed with 50 mL of 75 mL/L ethanol in distilled water. The 250 mL flask containing the slurry was covered with aluminum foil and placed in a water bath at 50 °C for 30 min. The samples were then cooled and 40 mL of 0.5 mol equi/L KOH were added. The slurry was kept under constant stirring at 200 rpm for 72 h. After this period, the alkali excess was titrated with 0.05 mol equi/L HCl, using phenolphthalein as an indicator. The solution was left to stand for 2 h and then any additional alkali, which may have leached from the sample, was titrated. A blank, using the original unmodified starch, was also used. The Ac % and the DS were calculated according to Equations (1) and (2) respectively.

$$Ac\% = \frac{\left(\left[V_{Blank} - V_{Sample}\right] \times Molarity of HCl + 0.043 \times 100\right)}{Sample weight}$$
(1)

Where: V_{Blank} is the titration volume used for the blank sample; V_{Sample} is titration volume used for each sample, both titration volumes were expressed in mL; the sample weight was expressed in g. DS is defined as the average number of sites per glucose unit that possess a (Whistler & Daniel, 1995).

$$DS = \frac{(162 \times Acetyl\%)}{(4300 - [42 \times Acetyl\%])}$$
(2)

2.4. Fourier Transform Infrared (FTIR) spectroscopy

The infrared spectra of the native and acetylated starches were obtained using a Fourier Transform Infrared (FTIR) spectrometer Prestige-21, Shimadzu, in the region of 4000–400 cm⁻¹. Pellets were created by mixing the sample with KBr at a ratio of 1:100 (sample:KBr). Ten readings were collected at a resolution of 4 cm⁻¹.

2.5. Pasting properties

The pasting properties of the starch samples (3.0 g, 14 g/100 g) moisture basis) were determined with a Rapid Visco Analyser (RVA-4; Newport Scientific, Warriewood, Australia). The viscosity was expressed in rapid visco units (RVU). The sample (3.0 g) was held at 50 °C for 1 min, heated to 95 °C in 3.5 min, and then held at 95 °C for 2.5 min. The sample was then cooled to 50 °C in 4 min and held at 50 °C for 2 min. The rotating speed was held at 960 rpm for 10 s and then maintained at 160 rpm during the process. Parameters including pasting temperature, peak viscosity, breakdown, final viscosity and setback were recorded.

2.6. Texture profile of gels

The gel texture profile was analyzed with Texture Analyser (TA.XTplus, Stable Micro Systems Ltd., Godalming, UK) according to the method described by Hormdok and Noomhorm (2007) with some modifications. After taking the RVA measurement, the gelatinized mixture in the canister was stored at room temperature ($20 \,^{\circ}$ C) for 24 h, allowing the formation of a solid gel. The canister was sealed with parafilm to prevent moisture loss during storage. The gel was punctured at 1.0 mms⁻¹ to a distance of 10.0 mm using a stainless steel cylindrical probe (P/20, 20 mm diameter). The peak force measured was reported as the gel hardness (height of the first peak).

2.7. Swelling power and solubility

The swelling power and solubility of the starches were determined as described by Leach, McCowen, and Schoch (1959).

2.8. Thermal properties

The gelatinization characteristics of starches were determined using differential scanning calorimetry (DSC model 2010, TA Instruments, New Castle, USA). Starch samples (approximately 2.5 mg, dry basis) were weighed directly in an aluminum pan, and Download English Version:

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