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Effect of conditions of modification on thermal and rheological properties of phosphorylated pumpkin starch

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ABSTRACT

This study aimed at analyzing the effect of conditions of modification process on thermal and rheological properties of phosphorylated pumpkin starch. The esterification process was conducted at 115 °C and 145 °C for 1, 2, and 3 h. The thermodynamic properties of samples were determined using differential scanning calorimetry (DSC), flow curves were plotted and the resulting curves were described the Herschel-Bulkley model, textural properties were evaluated with the TPA method. The data proved that the chemical modification of starch affected its rheological and thermal characteristics, but the direction and extent of the changes were found to depend on both temperature and duration of phoshorylation. The results demonstrated that temperatures of gelatinization of the samples modified at 145 °C were higher by 1.4-8.5 °C than those of the samples obtained at 115 °C. Prolongation of starch modification at 115 °C caused reduction of shear stress (from 2.10 Pa to 0.86 Pa), and higher temperature of esterification also reduced the value of this parameter. The hardness of the samples heated at 145 °C was higher by 45–59 N than that of heated at 115 °C. Adjustment of phosphorylation process caused an increase in gumminess by 1.8-37.9 N, wherein higher temperature and process prolongation resulted in the highest gumminess.
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1. Introduction

Starch is one of the biopolymers most widely used in the food industry [1,2], but it is also increasingly recognized as an ingredient in edible film [3–6] production or pharmaceutical industry. The major sources of starch are potato, corn, wheat, and rice [2]. But still, scientific articles have also been focused on new sources of starch like lotus seeds [7], litchi [8], canna [9] or pinhão [10]. The properties of new starches are analyzed because, generally, starch properties are dependent on its botanical origin. Starch is very popular among food manufacturers because it is easy to obtain and inexpensive, which makes it applicable in many branches of the industry. Starch can be used as a thickening, gelling and/or stabilizing agent, it may also be used to encapsulate flavors, but recently starch has been increasingly applied as a fat replacer [11–14].

Considering the growing attention paid to gluten-free diets, the gluten-free starch is more and more interesting for consumers and manufacturers. Pumpkin starch can be treated as an alternative source of starch. Structural and functional properties of starches from different kinds of pumpkin fruits have been studied [15–18]. Our experiments (publication in preparation) show that pumpkin starch is a good raw material for the production of edible foils because pumpkin films are much more transparent than the edible foils made of the commonly used starches.

However, native starch is susceptible to retrogradation and syneresis during storage, its functional properties are unstable and it is poorly resistant to changes in pH and temperature or to mechanical treatment. To avoid these undesirable changes, native starch is modified by physical or chemical methods [19–21]. The chemical modification of starch relies upon reactivity of two functional groups, amylose and amylopectin. One of the most useful modifications is phosphorylation which is conducted with phosphoric acid or salts [22–29]. Based on the way in which the acid molecule is incorporated, monostarch phosphate, distarch phosphate or their mixture can be distinguished. Pursuant to the EU Commission Regulation No. 1129/2011 L, phosphates belong to the first group of additives and should be added at *quantum satis*. According to the legal regulations, only low-substituted starch phosphate may be used in the food industry. Phosphorylated starches reduce syneresis and increase water binding capacity [30], but the direction and scope of these changes depend on the type of phosphorylation [27]. Phosphorylated starches are used as thickening agents in dressings, soups, sauces, instant whip, as ingredients of beverages or as calorie-reducing agents in margarine or meat

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products [12,13]. By using different technologies of starch modification, it is feasible to achieve different characteristics of the final product, hence selecting the appropriate modification process is vital in food production. The use of so modified starch should confer desirable properties to the manufactured products, without hindering the production processes. Therefore, it is important that the selection of starch takes into account factors related to nutrition, production, distribution, and marketing.

Despite there being articles on various kinds of pumpkin starches [15–18], information is still lacking about rheological and thermal properties of phosphorylated pumpkin starch. The knowledge about these properties of phosphorylated pumpkin starches may contribute to the effective reduction in the quantity of starch added to food products and, at the same time, allow reducing production costs and making this kind of product a healthier and safer option for consumer health.

Since the selection of appropriate conditions for the esterification process determines the functional properties of modified starch preparations, the aim of this study was to analyze the influence of the temperature and duration of the phosphorylation process of pumpkin starch on its selected rheological properties.

2. Material and methods

2.1. Material

The experimental material consisted of starch isolated from pumpkin fruits *Cucurbita maxima* var. Amber. The pumpkin fruits were obtained from the Experiment Station in Mydliniki, Department of Plant Protection, University of Agriculture in Kraków, Poland.

The reagents used during modification were pure for analyses. Sodium tripolyphosphate (STPP) and sodium trimetaphosphate (STMP) were purchased from Sigma-Aldrich, whereas Na_2SO_4, HCl and NaOH were purchased from POCh, Poland.

2.2. Starch isolation

Starch was isolated from pumpkin fruit according to Moreira et al. [31] method with minor changes. Ripe pumpkins were harvested and peeled, cut into pieces and ground. The obtained pulp was soaked in distilled water at room temperature. The suspension was placed on a nylon sieve (100 μm mesh) and thick slurry was rinsed with distilled water. The dough was continuously kneaded and rinsed. The resulting starch milk was poured into 200 mL plastic centrifuge vessels and centrifuged at 4000×*g* for 20 min. After centrifugation, the supernatant was decanted and the layer of impurities was removed with a spatula. A new portion of starch milk was added and the sample was mixed and centrifuged again. The process was repeated three times, after which the supernatant and impurities were removed, distilled water was added, and the sample was mixed and centrifuged again. After the final centrifugation, the supernatant was decanted, the impurities were removed and the sediment was transferred to plastic dishes, ground and air-dried at room temperature while being constantly stirred. After drying, the starch was ground in a Retsch RM-200 mortar and filtered through a 125 μm Retsch AS-200 sieve. The obtained starch was characterized by 14.88% moisture, 97% starch in dry matter, 0.4% protein in dry matter, 0.15% fat in dry matter, and 0.026% phosphorus in dry matter.

2.3. Phosphorylation

The pumpkin starch was phosphorylated according to a modified procedure of Lim and Seib [32], as described by Rożnowski et al. [27]. Starch modification began with the melting of 15 g of sodium tripolyphosphate (STPP) and 6 g of sodium trimetaphosphate (STMP) with 300 mL of deionized water with 15 g Na_2SO_4. The solution was mixed and pH was adjusted to 9.5 by using 10% (w/w) solutions of HCl or 10% (w/w) solution of NaOH. That solution was added to 300 g of starch (dry matter) and the obtained suspension was mixed, and pH was adjusted to 9.5. by using 5% solutions of HCl or NaOH. The suspension was mixed at room temperature for 1 h, and transferred quantitatively onto a Petri dish and dried at 40 °C in a drier to a moisture content of approximately 10%. The resultant samples were ground in a Retsch RM200 grinder and sieved through Retsch AS200 screens with a mesh diameter of 125 μm. The resultant starch was divided into 7 portions and dried at a temperature 115 and 145 °C for 1, 2 or 3 h. After drying, the samples were cooled in vacuum desiccators and mixed with 1200 mL of distilled water, and pH was again adjusted to 6.5 by using 5% solutions of HCl or NaOH and excess solution was mixed again with an electric stirrer at 3700 rpm for 15 min. Next, 750 mL of distilled water were added to the excess sediment and the sample was again mixed with an electric stirrer at 3700 rpm for 15 min. This procedure was repeated two more times and the obtained samples were air-dried to a moisture content of approximately 10%, and ground in a Retsch RM200 grinder and sieved through Retsch AS200 screens with a mesh diameter of 125 μm. However, the sample prepared at 145 °C for 3 h could not be obtained because it was totally soluble in water during preparation process. The phosphorus content in pumpkin starch phosphorylated at 115 °C for 1 h reached 0.295%, at 115 °C for 2 h reached 0.386%, at 115 °C for 3 h reached 0.478%, at 145 °C for 1 h reached 0.519%, and at 145 °C for 2 h reached 0.506%.

2.4. Gelatinization properties

Gelatinization temperatures of starches were measured with a differential scanning calorimeter (DSC 204F1 Phoenix, Netzsch GmbH, Germany) according to the method [28,29]. In order to determine the thermal properties of the samples, 3.5 mg of the sample and 10.5 mg of water were placed in calorimetric aluminum pans, sealed hermetically and stored for 24 h at room temperature. The samples were then heated from 20 to 110 °C at a rate of 10 °C/min. After analysis, the pans were stored at 4 °C for 7 days, after which the thermodynamic characteristics of retrogradation were determined using a similar procedure. An empty pan was used as the reference. The gelatinization and retrogradation endothermic peaks were characterized by onset (T_o), peak (T_p) and endset (T_e) temperatures as well as enthalpy values. Additional calculations were made for the percentage ratio of retrogradation enthalpy to gelatinization enthalpy.

2.5. Determination of flow curves

Flow curves of the pastes obtained from native and phosphorylated starch were plotted with the use of a Rheolab MC1 rotary rheometer (Physica Meβtechnic GmbH, Germany), applying a measuring system of coaxial cylinders Z3 DIN: bob diameter – 25.00 mm, cup diameter – 27.12 mm. The rheometer and thermostat (Viscotherm2) were computer-controlled using US 200 software (Physica Meβtechnic GmbH, Germany). To determine flow curves, 2% (w/w) solutions of starches in water were prepared.

The samples were blended at room temperature for 15 min with a mechanical stirrer (300 rpm). The solutions were then heated in a water bath at 95 ± 1 °C for 30 min with continuous stirring (300 rpm). The resulting pastes were immediately placed in the measuring element of the rheometer and kept at 50 ± 0.5 °C for 10 min before analysis. The flow curves were plotted according to the following program: shearing rate increase from 1 to 500 s^{-1} within 3 min, constant shearing rate of 500 s^{-1} for 2 min, shearing

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