Food Chemistry 245 (2018) 385–393

Contents lists available at ScienceDirect

Food Chemistry

journal homepage: www.elsevier.com/locate/foodchem

Impact of hydrothermal modifications on the physicochemical, morphology, crystallinity, pasting and thermal properties of acorn starch

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ARTICLE INFO

Keywords:
Acorn starch
Hydrothermal modification
Morphology
Structure
Functional properties

ABSTRACT

Native acorn starch had high purity and the granules were mainly elliptical and spherical with the mean diameter of 7.32 μm. Hydrothermal modifications slightly changed the morphology. The solubility, swelling properties and amylose leaching of acorn starch were mostly influenced by Heat-moisture treatment (HMT). XRD pattern of native starch (C-type) did not change on hydrothermal modifications, but native and annealing (ANN) modified starches showed the most crystallinity. DSC results showed that the gelatinization temperatures and enthalpy of native starch were 59.9, 71.3, and 80.6 °C and −14.9 mJ/mg, respectively, and hydrothermal treatments generally increased the gelatinization temperatures. Regarding to RVA results, peak, breakdown, trough, setback, and final viscosities of native starch were 415, 143, 272, 168, and 440 RVU, respectively, and viscosity parameters of native starch were mainly more than those of hydrothermally modified starches. Generally, the intensity of the effects of hydrothermal modifications followed the order: HMT > dual modifications > ANN.

1. Introduction

It is essential to find local and valuable sources of starch (like acorn) which are comparable with commercial ones (corn and wheat), and can be used to produce many other products. Acorn is the fruit of oak trees which belong to the genus *Quercus*, more than 300 species of which are distributed in Eurasia, North and Central America and in North Africa (Toumi & Lumaret, 2001). *Quercus brantii*, named Persian oak or the Zagrossian oak, is the dominant species grows in the Zagros mountain chain in Iran, in an area of about 4 million hectares (Saffarzadeh, Vincze, & Csapó, 1999).

Acorns contain significant amount (about 58%) of starch (Saffarzadeh et al., 1999) and have been used in different foods (Aee, Hie, & Nishinari, 1998; León-Camacho, Viera-Alcaide, & Vicario, 2004; Molavi, Keramat, & Raisee, 2015). In the past, due to the deficiency of wheat, acorn flour was used for making a kind of bread called “Kalg” in the Zagrossian region. But, its production is low today because of its lower quality (lack of gluten) in comparison with wheat bread. Therefore, nowadays the trees are underexploited for the production of charcoal and the fruits are left to rot or to a lesser extent, are used as animal feed. Thus, the idea of extracting starch from the acorn provides us with some advantages as follows: preserving oak forests; motivating to set up an acorn starch production factory with high profitability because these forests are natural and do not require agricultural practices and the fruits should be just gathered when they are ripe; and increasing in the potential of starch production of the country by introducing a new source of starch. Furthermore, the by-products (like tannins, tannic acid, and oil) are also valuable (León-Camacho et al., 2004; Tejerina, García-Torres, Vaca, Vázquez, & Cava, 2011). Other researchers have also mentioned the benefits of starch extraction from acorns (Correia & Beirão-da-Costa, 2010; Stevenson, Jane, & Inglett, 2006).

Since native starch is unable to meet all the demands in food and non-food industries, it should be modified by various methods. It has recently been a growing trend toward treating starch with hydrothermal methods like heat-moisture treatment (HMT) and annealing (ANN), which modify the properties and structure of starch without any damage to granular integrity. HMT involves heating starch at high temperatures while moisture is low, but ANN refers to heating a slurry of starch (with excess water) below its gelatinization temperature. Both treatments lead to structural changes within the amorphous and crystalline regions to different extents (Chung, Hoover, & Liu, 2009). The properties of hydrothermally treated starch depend greatly on two main factors: the conditions of treatments (i.e. moisture content, temperature, duration, etc.) and the source of starch (i.e. its physicochemical properties). So, for each source of starch, the effects of these treatments are different and should be discovered individually. Dual modifications (chemical or physical) have also been of great interest to many

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http://dx.doi.org/10.1016/j.foodchem.2017.10.117
Received 23 July 2017; Received in revised form 21 October 2017; Accepted 23 October 2017

researchers. There are few publications on dual hydrothermal modification of starch, especially those considering the sequence of treatments. On the other hand, the effects of dual modifications on the properties of various sources of starch have been different and in some cases contradictory (Chung, Liu, & Hoover, 2010; Chung, Hoover, et al., 2009). Consequently, many investigations should be conducted to reveal the importance of each effective factor and the extent of changes for different sources of starch. On the other hand, since chemicals and genetic modification may not be permitted to change the properties of existing commercial starches (Chung, Hoover, et al., 2009), research on hydrothermal treatments is crucial.

Hydrothermally modified starches own various applications in food industry; such as in canning and frozen foods (Jayakody & Hoover, 2008); pouch packed, and resistant starch production (Chung, Liu, & Hoover, 2009); fat replacer technology (Jayakody & Hoover, 2008); and biodegradable films (Molavi, Behfar, Shariati, Kaviani, & Atarod, 2015). Since there was not much information about Persian acorn starch, the aims of this research were (i) to determine physicochemical properties of the native Persian acorn starch in order to introduce its potential applications in the future and (ii) to investigate the effects of single and dual hydrothermal treatments (HMT, ANN, HMT-ANN, and ANN-HMT) on its properties because these treatments broaden the applications of starch without using any chemicals, so the resulting modified starch will be GRAS. In this paper, we focused on the compositions, color, morphology, swelling properties, amylose leaching, crystallinity, gelatinization, and pasting properties of native and hydrothermally modified starches.

2. Materials and methods

2.1. Preparation and extraction procedure

Acorns (*Q. brantii*) were provided from Lordegan region, Chaharmahal va Bakhtiari Province, Iran, at the maturity stage and kept at a cool, dark place until the experiments began. The preparation, milling and extraction procedures were as follows: (1) pre-drying at 40 °C/24 h in an oven (Memmert UNB 500, Germany), (2) peeling and cutting into small pieces, (3) final drying at 40 °C in the same oven, (4) milling the pieces using a hammer mill and passing the flour through a 1 mm sieve, (5) starch extraction using the low shear, alkaline pH, and successively three sieves (LSA3S) method proposed by Correia and Beirão-da-Costa (2012). Starch extracted by this method has been shown to have high purity and yield and its physical and chemical properties are less affected. The isolated starch was dried overnight in the aforementioned oven at 40 °C.

2.2. Hydrothermal treatments

2.2.1. Heat-moisture treatment (HMT)

Native starch was weighed into glass containers and its moisture content was adjusted to 20% by adding the appropriate amounts of distilled water. After sealing, the containers were kept at room temperature for 24 h and then heated at 110 °C/24 h in the oven. Then, the containers were opened and the treated starch samples were dried to uniform moisture content (~10%).

2.2.2. Annealing (ANN)

Acorns slurry (70% moisture) was heated at 50 °C/24 h in a water bath (Memmert WNB 29, Germany) and then centrifuged (Sigma, Germany) at 2000*g* for 10 min and the supernatant was discarded. Next, the annealed starch was washed once with deionized water and finally dried at 40 °C in the aforementioned oven.

2.2.3. Dual modifications (HMT-ANN and ANN-HMT)

For HMT-ANN, heat-moisture treated starch (Section 2.2.1) was subjected to annealing treatment (Section 2.2.2) and for ANN-HMT, annealed starch (Section 2.2.2) was subjected to heat-moisture treatment (Section 2.2.1).

2.3. Chemical compositions measurement

Moisture, ash, and nitrogen contents of starch samples were determined according to the AACC methods (AACC, 2000). Free and bound lipids were analyzed by the method of Vasanthan and Hoover (1992). Apparent amylose content was measured by the colorimetric method (Williams, Kuzina, & Hlynka, 1970).

2.4. Color analysis

Color of starch samples were assessed using a HunterLab spectrophotometer (FMS Jansen, USA). First, the instrument was calibrated using black and white tiles. Then L^* (lightness), a^* (red/green opponent colors), and b^* (yellow/blue opponent colors) of starch samples were measured (10 readings for each replication). Finally, the color-difference (ΔE), compared to native starch, was calculated by using the Eq. (1).

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (1)$$

2.5. Scanning electron microscopy (SEM)

Granule morphology of the native and modified starch samples were examined using a Zeiss (evo, Germany) scanning electron microscope. Dry Starch samples were placed on circular aluminum stubs with double-sided carbon adhesive tape and then coated with a thin film of gold using a sputter coater (Emitech, UK). The accelerating potential was 13 kV. The images were captured at different magnifications and analyzed with the ImageJ software (ver. 1.49, National Institutes of Health, USA) in order to determine the size of granules.

2.6. Swelling power, solubility, and amylose leaching measurement

Swelling power, solubility and amylose leaching of starch samples (at 90 °C) were determined according to the methods described by Tattiyakul, Naksriarporn, Pradipasena, and Miyawaki (2006). For this purpose, starch (0.5 g, dry base) was dispersed in water (15 ml). The dispersion was heated and mildly agitated at 90 °C for 30 min. The dispersion was then centrifuged at 3000*g* for 15 min. Afterward, the supernatant was separated and dried at 100 °C until reaching a constant weight (m_s). The swelling power and solubility were calculated according to the following equations:

$$\text{Swelling power [g/g dry starch]} = \frac{m_{sw}}{m_0(1 - solubility)} \quad (2)$$

$$\text{Solubility [g/g dry starch]} = \frac{m_s}{m_0} \quad (3)$$

where, m_0 is the amount of dry starch and m_{sw} is the weight of swollen starch granules.

For measuring amylose leaching, 60 mg sample was dispersed in 15 mL distilled water in a centrifugal tube and heated at 90 °C for 30 min. Afterward, the gelatinized dispersion was centrifuged at 3000*g* for 10 min. Then the supernatant was decanted and its amylose content was measured according to Williams et al. (1970). Amylose leaching was calculated by the following equation:

$$\text{Leached amylose [mg/100 mg amylose]} = \frac{m_{la}}{m_a} \times 100 \quad (4)$$

where, m_{la} is the weight of leached amylose and m_a is amylose content in 60 mg starch.