



Characterization of acid hydrolysis of granular potato starch under induced electric field



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ABSTRACT

Compared to traditional heating methods by static immersion or stirring, induced electric field (IEF) treatment can significantly increase hydrolysis rate of potato starch. The aim of this study was to examine the characterization of acid hydrolysis of granular potato starch under IEF. Potato starch was hydrolyzed using 0.15 mol/L HCl combined with IEF (IEF-HCl) at an excitation voltage of 75 V from 4 h to 120 h. The changes in granular, crystal, and molecular structure of starch granules during IEF-HCl hydrolysis were investigated. Scanning electron microscope analysis showed the outer surface of potato starch was firstly attacked by free ions and then the inner structures was completely disrupted and broken into pieces. X-ray diffraction studies indicated that the crystallinity decreased from 38.92% to 34.9% after 4-h hydrolysis, and then gradually increased to 41.88% as hydrolysis time increased to 96 h, but decreased thereafter. The weight-average molecular weight dramatically decreased from 104.7×10^6 Da to 9.291×10^6 Da in the first 24 h and then decreased gradually. These results suggested that there were three steps in the catalytic process of IEF-HCl hydrolysis. Initially, hydrogen ions destroyed the external crystalline shell of granules. Next, hydrogen ions attacked the loose amorphous region and dense crystalline part within starch granules simultaneously. Finally, the remaining crystalline region was hydrolyzed slowly.

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1. Introduction

Starch, which is the most abundant storage polysaccharide in plants, has been widely utilized in food industry for its functionalities in thickening, stabilizing, texturizing, gelling, film forming, and encapsulation (Singh, Chawla, & Singh, 2004; Zhang, Tao, Niu, Li, & Chen, 2017). However, native starches have limited industrial applications due to their low shear stress resistance, poor thermal stability, susceptibility to retrogradation, and large granule size causing facile break (Hong, Chen, Zeng, & Han, 2016). Acid hydrolysis can modify starch granular structure and produce thin-boiling

starch for food, pharmaceutical, and chemical industries (Wang & Copeland, 2015). Traditionally, acid hydrolysis of starch is performed by dilute sulfuric acid (H_2SO_4) or hydrochloric acid (HCl) at a temperature below starch gelatinization temperature for different duration (Wang & Wang, 2001). The process exhibits various advantages including relatively low temperature, simple pretreatment for starch feedstock, and inexpensive acid catalyst. Because of the semi-crystal structure, almost starches exhibit two hydrolysis stages: the first stage with relatively fast rate corresponds to hydrolysis of amorphous part within starch granules and the subsequent slow hydrolysis of crystalline lamellae occurs during the second stage (Wang & Copeland, 2015; Wei et al., 2013). The rigid crystalline lamellae prevent the penetration of hydrogen ions and the transformation of glucopyranose rings from chair to half-chair conformation, causing a long acid hydrolysis process (5–15 days) under mild condition (Corre, Bras, & Dufresne, 2010).

Non-conventional techniques including microwave heating (Li et al., 2001; Yu, Chen, Suree, Nuansri, & Wang, 1996), high

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hydrostatic pressure (Lee et al., 2006), ultrasound (Kim, Han, Kweon, Park, & Lim, 2013), and high-pressure homogenization (Wei, Cai, Jin, & Tian, 2016) have been applied to accelerate acid hydrolysis of biomass. Recently, the application of ohmic heating (OH), moderate electric field (MEF) and pulsed electric field (PEF) in food industry has gained attention. Electrical techniques possess a large number of potential use for extraction (Vorobiev & Lebovka, 2013), pickling (Jin et al., 2015), film preparation (Souza et al., 2010), pasteurization (Kaur & Singh, 2016), protein gelation (Rodrigues et al., 2015), blanching (Sakr & Liu, 2014) and organic synthesis (Cardoso et al., 2015). Studies have been reported on physical properties of starch granules treated by OH and PEF. Thermal characteristics, such as gelatinization temperature and enthalpy, of OH-modified rice starch and rice flours were changed because the samples were gelatinized during treatment (An & King, 2007). Application of OH with higher voltage was more effective to gelatinize jicama and cassava starches, and then starches with various degrees of gelatinization can be obtained by controlling OH conditions (Martínez-Bustos, López-Soto, Zazueta-Morales, & Morales-Sánchez, 2005). The adjustment of electrical conductivity (σ) of starch suspension during OH was developed to analyze starch gelatinization process (Li, Li, Li, & Tatsumi, 2004; Wang & Sastry, 1997; Wong, Xu, Lu, & Li, 2011). PEF dissociates and damages granular starches (Han, Zeng, Yu, Zhang, & Chen, 2009; Han, Zeng, Zhang & Yu, 2009), as well as assists the acetylation of cassava and potato starch (Hong et al., 2016; Hong, Zeng, Buckow, Han & Wang, 2016). OH and PEF exhibit enhanced mass transfer effects, suggesting the possibility to enhance reactive dynamics. However, these electrotechniques are severely restricted by adverse electrochemical reaction due to the insertion of excited metal electrode in conductive media.

Therefore, induced electric field (IEF) method has been developed based on a transformer with coils and magnet circuit for the varying field generation. In IEF, flowing electrolyte solution is used as the secondary coil (sample coil). When excitation voltage is applied on primary coil, an alternating IEF emerges in the sample coil, resulting in rapid electromigration of charged ions that accelerate mass transport and enhance reactivity. Our previous study suggested that IEF treatment significantly affects the structural and physicochemical properties of potato starch (Li et al., 2016). However, IEF-assisted HCl (IEF-HCl) hydrolysis process of potato starch has not been systematically investigated. Therefore, the study aimed to explore the mechanism of IEF-HCl hydrolysis of potato starch by determining their granular, crystal, and molecular structure.

2. Materials and methods

2.1. Materials

Potato starch (amylose content 15.5%, moisture content 9.6%) was produced by the Dutch Meelunie Company (Amsterdam, Holland). Potassium chloride, hydrochloric acid, potassium sodium tartrate, 3,5-dinitrosalicylic acid, phenol, sodium sulfite, sodium nitrate, sodium hydroxide, glucose, and other chemicals were of analytical grade and were purchased from Sinopharm Chemical Reagent Co., Ltd. (Suzhou, China).

2.2. IEF-assisted acid hydrolysis

The IEF system was developed on the basis of transformer concept and its operational principle as well as the schematic diagram have been reported in our previous study (Li et al., 2016). Potato starch (120 g) and potassium chloride (12 g) were added into 400 mL 0.15 mol/L HCl solution to obtain a starch suspension (30%

w/v). The suspension was then pumped into the system by a peristaltic pump (BT100S-1, Lead Fluid Technology Co., Ltd., Shanghai, China). Once an excitation voltage generated by power source (PS-6105, Taiwan Pusi Electronics Co., Ltd., Taiwan) is imposed on primary coils, contributes to an alternating magnetic flux, and therefore an alternating induced voltage occurs in secondary coil (sample coil) to facilitate IEF processing. The output voltage in sample coil was measured using a pair of platinum electrodes connected to an AC millivolt meter (UT631, Uni-Trend Group Limited Co., Ltd., Hong Kong, China) (Yang et al., 2015). IEF treatment was performed under the conditions: excitation voltage at 75 V; frequency at 400 Hz; flow rate at 300 mL/min; reaction temperature at 45 °C, reaction time ranged from 4 h to 120 h. The low excitation voltage used in this study was to slow the hydrolysis process, thus allowing to exactly analyze the mechanism of IEF-HCl hydrolysis of potato starch. The reaction was stopped by adding 2 mol/L NaOH to adjust the suspension to pH 7.0. After cooling to room temperature, the reaction mixture was centrifuged at 1600 g for 10 min. The reducing sugar content in supernatant was determined. And the precipitate was washed four times with distilled water (200 mL each time) and dried in an air oven at 40 °C for 24 h. After drying, samples were passed through a 120-mesh sieve for subsequent analysis. Potato starch treated by static immersion at 45 °C and magnetic stirring at 45 °C, were defined as controls.

2.3. Reducing sugar content

The reducing sugar content in supernatant was determined by using DNS reagent with D-glucose as a standard. The test was performed with 2 mL DNS reagent which was mixed with 1 mL supernatant and 1 mL distilled water in capped test tubes. The mixture was heated at boiling water for 5 min to develop red brown color, and then immediately cooled to room temperature in an ice-water bath. The absorbance was recorded spectrophotometrically at 540 nm by using a V-1800 spectrophotometer (Mapada, Shanghai, China). The reducing sugar content was calculated based on the standard curve obtained with glucose. To prepare DNS reagent, 6.3 g 3,5-dinitrosalicylic acid, 262 mL 2 mol/L NaOH solution, 185 g potassium sodium tartrate, 500 mL distilled water, 5 g phenol, and 5 g sodium sulfite were mixed and stirred at 50 °C until dissolution. After cooling to 25 °C, the solution was transferred to a 1000 mL volumetric flask and then distilled water was added to reach the necessary volume. The prepared solution was stored in a brown bottle at 4 °C for 7 days prior to use.

2.4. Scanning electron microscopy (SEM)

The morphology of starch granules was observed using a scanning electron microscope (SU 1510, Japan) with an accelerating voltage of 5.00 kV. For observation, samples were fixed on an aluminum specimen holder with double-sided adhesive tape followed by coating with gold under vacuum evaporation.

2.5. Particle size distribution

The particle size distribution of native and modified potato starches was examined by a particle size analyzer (Microtrac S3500, Montgomeryville, USA) using dry method. The measurement was performed under following conditions: weight of sample about 0.3 g; injection rate 50%; dispersed gas air; gas pressure 5×10^4 Pa; run time 30 s. The D (4, 3) represents volume mean diameter and the D (3, 2) represents area mean diameter. The criterions D10, D50, and D90 are particle sizes at which 10%, 50%, and 90% of all particles by volume were smaller, respectively.

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