



Multiple endothermic transitions of acid hydrolyzed and heat-moisture treated corn starch



Jun-jie Xing^a, Dong Li^{a, *}, Li-jun Wang^{b, **}, Benu Adhikari^c

^a Beijing Advanced Innovation Center for Food Nutrition and Human Health, College of Engineering, National Energy R & D Center for Non-Food Biomass, China Agricultural University, P. O. Box 50, 17 Qinghua Donglu, Beijing 100083, China

^b College of Food Science and Nutritional Engineering, Beijing Key Laboratory of Functional Food from Plant Resources, China Agricultural University, Beijing, China

^c School of Applied Sciences, RMIT University, Melbourne City Campus, VIC 3001, Australia

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ABSTRACT

The multiple thermal transitions of acid-hydrolyzed and heat-moisture treated corn starch samples in excess water were measured using differential scanning calorimetry (DSC) and interpreted using a polymer swelling and dissolution theory. The dual treatment of acid hydrolysis (AH) and heat-moisture treatment (HMT) with different extent of reaction generates starches with various degrees of residual crystalline structure. Multiple endothermic transitions (G and M1) were firstly observed in DSC traces even in excess water. In this study, the G and M1 endotherms were proposed to be related with the energy consumption associated with granule swelling and crystallites dissolution respectively. In contrast, only a single M1 endotherm transition from a broad to a sharp and narrow endotherm upon HMT was consistently observed for starch samples acid hydrolyzed for 48 and 72 h, which testified the dominance of rearrangement over the destruction upon HMT. This novel theory describing the multiple phase transition was also evidenced by the variation of swelling power and relative crystallinity.

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1. Introduction

A proper understanding of starch gelatinization is essential for creating desired texture in cereal-based food products. Gelatinization has been broadly defined as a swelling driven process (Donovan, 1979) or an order-disorder phase transition (Hoover, Hughes, Chung, & Liu, 2010). When heated in excess water, starch granules undergo swelling, lose their birefringence, and convert themselves into a colloidal dispersion (Ratnayake & Jackson, 2009). This definition implies that at least three distinct changes occur during the gelatinization of starch: absorption of water and swelling of granules, disruption of crystalline structure, and dissolution of starch chains (Wang, Li, Yu, Copeland, & Wang, 2014). Generally, these thermal behaviors can be quantitatively studied by differential scanning calorimetry (DSC). There have been various explanations on these thermal transitions when heating starch granules under excess water (Li, Xie, Yu, & Gao, 2013; Ratnayake &

Jackson, 2006) and insufficient water conditions (Fukuoka, Ohta, & Watanabe, 2002; Jiranuntakul, Puttanlek, Rungsardthong, Panchanron, & Uttapap, 2011; Pinto et al., 2012; Pukkahuta, Suwannawat, Shobsngob, & Varavinit, 2008). However, the explanation of the nature of endothermic and exothermic phenomena during starch gelatinization in excess water is still debatable.

There are several theories available in literature to describe the biphasic G and M1 endothermic transitions observed in DSC thermograms, including: water availability (melting of crystallites driven by swelling of amorphous regions and then melting of the remaining crystallites), crystallite stability (melting of the least stable crystallites with subsequent progressive melting of more stable crystalline domains), sequential phase transition (first amorphous and then crystalline regions), three-stage phase transition (partial melting, recrystallization, and total melting) (Ratnayake & Jackson, 2007, 2009). However, none of the above theories is able to adequately explain starch gelatinization process and it needs further consummate on the theory to interpret the complicated phase transitions presented on the DSC curves especially in excess water. Recently, a new perspective of the swelling and dissolution of the polymer was proposed by Wang and Copeland (2012a) to interpret the nature of thermal transition of

* Corresponding author.

** Corresponding author.

E-mail addresses: dongli@cau.edu.cn (D. Li), wj@cau.edu.cn (L.-j. Wang).

acid-hydrolyzed pea starch. This work provided new insights into the gelatinization of starch. After studying the swelling behavior of different starch granules over a wide range of water content, Wang et al. (2014) have well explained the multiple DSC thermograms with this novel polymer swelling and dissolution theory. As argued earlier, no theory or model adequately explains the DSC endotherms observed at different water contents. Therefore, this theory is very appealing because it explains the mechanism of gelatinization in the much wider spectrum of water contents rather than proposing different mechanisms, for example, for high water contents.

On the other hand, the difficulty in explaining the mechanism of starch gelatinization is at least partly due to the structural complexity of the amorphous and crystalline regions of the overall starch granules (Fu, Wang, Zou, Li, & Adhikari, 2014). These complex structures of native starches make it difficult to figure out the details of starch gelatinization during which the changing process are intertwined together and there is no clear boundary between them (Fukuoka et al., 2002). Based on this, maybe the gelatinization process could be simplified if we could find a way to clarify the molecular and granular structure and to control the part and composition of granules that actually gelatinized. In our previous study, we found that the dual treatment with acid hydrolysis (AH) and heat-moisture treatment (HMT) in AH-HMT sequence can impart the starches with different crystalline structure and thermal stability (Xing, Liu, Li, Wang, & Adhikari, 2017). These starches with different microstructure will be carefully prepared as the test samples in this study. Thus, there were two objectives to accomplish. Firstly, to investigate the progressive changes in swelling behavior and crystalline structure of AH starch subjected to HMT. Secondly, to interpret the thermal transitions of AH-HMT starch samples in excess water by using polymer swelling and dissolution theory. We believe that this work will provide valuable information since the application of swelling and dissolution theory will provide greater insight into the phase transition behavior of starch under excess water conditions. Also, the combined AH-HMT procedure produces starch samples with different degree of crystallinity and swelling power, and judicious control of these two is important in product development.

2. Materials and methods

2.1. Materials

Corn starch was obtained from Hebei Zhangjiakou Yujing Food Co., Ltd. (Hebei, China). Hydrochloric acid and sodium hydroxide were provided by Beijing Chemical Company (Beijing, China) and were of analytical grade. Deionized water was used throughout the work.

2.2. Preparation of AH-HMT starch samples

The native corn starch was subjected to AH and HMT in AH-HMT sequence according to our previous work with slight modification (Xing et al., 2017). The starch was firstly acid treated for 6, 12, 24, 48, 72 h and then heat-moisture treated for 15, 30, 60 min at 120 °C. These samples are abbreviated as AH_{6, 12, 24, 48, 72}-HMT_{15, 30, 60} in this study.

About 60 g starch was dispersed in 1 mol/L hydrochloric acid to produce suspensions with 300 g/L starch slurry. These suspensions were transferred to an incubation chamber maintained at 40 °C. To ensure a more homogeneous hydrolysis, these suspension samples were continuously stirred while they were incubated. After

incubation for 6, 12, 24, 48, 72 h, the slurries were individually taken out and immediately centrifuged at 6000×g for 15 min. The solid mass obtained after centrifugation was neutralized (pH 7) using 40 g/L NaOH and then centrifuged once again at 6000×g for 15 min. The solid mass obtained after second centrifugation was washed using distilled water and finally vacuum-filtered. These washing and vacuum filtration steps were repeated three times. These starch samples were air-dried at 40 °C.

The AH samples obtained following the above-mentioned protocol were then subjected to the HMT procedure. The moisture content of AH samples was determined, recorded, and then adjusted to 0.3 g/g by spraying a calculated amount of distilled water. These moistened starch samples were sealed and equilibrated in Duran glass containers for 24 h at ambient temperature and then placed in a forced air oven maintained at 120 °C for 15, 30, and 60 min. After incubation, the samples were cooled to ambient temperature. These samples were further air dried at 40 °C. The dried samples were then ground to pass through a 120 mesh sieve. Finally, the AH-HMT starch samples were packed in zipper bags and kept in desiccators until used.

2.3. Determination of swelling power and solubility

About 0.3 g was weighed and transferred into a centrifuge tube having its own screw cap. Deionized water was added to obtain 10 g/L starch concentration and the cap was put in place. This starch suspension was heated to 90 °C using a shaking water bath and this temperature was maintained for 30 min. The fully swelled sample was cooled to ambient temperature and centrifuged at 1008×g for 15 min. The supernatant was carefully separated from the sediment and dried at 110 °C until the mass was constant. The mass values of both wet residue and dried supernatant were also measured. The swelling power (g H₂O absorbed/g dry starch) and solubility (% of dry starch) of the samples were calculated using equations (1) and (2):

$$\text{Swelling power (SP)} = \frac{[\text{mass of wet residues}]}{\times [\text{mass of dried sample}]} \quad (1)$$

$$\text{Solubility (SO)} = \frac{[\text{mass of dried supernatant}]}{\times [\text{mass of dried sample}] \times 100} \quad (2)$$

The amount of water bound per anhydroglucose unit (WB/AGU) in the residues was also calculated according to the methods reported by Wang and Copeland (2012b), from the knowledge of moles of water absorbed per mole of anhydroglucose unit as given by:

$$\text{WB/AGU} = \frac{[\text{mass of water in residue}]}{\times [\text{mass of starch in residue}] \times [162/18]} \quad (3)$$

2.4. Determination of relative crystallinity

The crystallinity of starch granules was measured by using an X-ray diffractometer (XD-2, Beijing Purkinje General Instrument Co., Ltd., China). A nickel-filtered CuK α ($\lambda = 0.15406$ nm) radiation at a voltage of 36 kV and a current of 20 mA was used in these tests. Samples were scanned from 10° to 40° (2 θ) with a scanning rate and sampling interval of 1°/min and 0.02°, respectively. The relative crystallinity (RC) values were calculated by using MDI Jade 5 software (Materials Data Inc., Livermore, CA).

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