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Temperature thresholds and time-temperature dependence of gelatinization for heat-moisture treated corn starch

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ABSTRACT

The time and temperature dependence at different stages of starch gelatinization has been poorly understood. In order to fill this gap in knowledge, we employed heat-moisture treated corn starch and developed a novel gradient gelatinization method with short (5 min) and long (20 and 30 min) isothermal holding within 64–95 °C to determine the relevant temperature thresholds. The increase in viscosity values (Δμ-values) during 5 min isothermal holding demonstrated the temperature-dependent behavior of corn starch gelatinization. The time-dependent nature of which was also evidenced with long-term heat-holding method. 70 and 76 °C were proposed as the two critical temperature thresholds TTS_1 and TTS_2, which were associated with amorphous swelling and disintegration of crystalline domains, respectively. Furthermore, the individual influence of time and temperature at different gelatinization stages were discussed based on a multi-stage gelatinization theory. The degree of time-dependence was different from different gelatinization stages occurring in between TTS_1 and TTS_2.

1. Introduction

Gelatinization is one of the most important and commonly observed physicochemical and functional properties of starch (Li et al., 2013; Schirmer et al., 2015). Many starch-based food formulations require starch with different gelatinization degree for improved product characteristics. Design of process flow charts and processing conditions of starch or starch-containing foods has to consider the nature of gelatinization of starch including isothermal or non-isothermal modes of heating (Ahmed, 2012a; Malumba et al., 2013; Ratnayake and Jackson, 2006). Although the starch gelatinization has long been studied, the time and temperature dependence of gelatinization is still not fully understood.

Many research endeavors have focused on quantifying, modeling, and predicting the kinetics of starch gelatinization in terms of gelatinization temperature, gelatinization enthalpy, and rheological viscosity (Anuntagool et al., 2017; Pérez-Santos et al., 2016; Spigno and De Faveri, 2004). As early as 1980, Bakshi and Singh (1980) have studied the influence of time and temperature on the gelatinization of rough and brown rice starch using isothermal treatment method. Wirakartakusumah (1981) observed that the lag time at different temperatures plays a key role in determining the degree of gelatinization, the aspect was not taken into consideration by previous authors. The temperature and heating time dependency of cross-linked wheat starches have been investigated by measuring the granular diameter of swelling starches at temperatures ranging from 30 to 90 °C (Choi and Kerr, 2004), showing the importance or the impact of time to the swelling process is different at different stages. Recently, Carlstedt et al. (2015) found that the swelling of starch granules required several minutes to complete even at high temperatures. Pérez-Santos et al. (2016) introduced an empirical parameter (C_∞, quantifies the extent of gelatinization at infinite time and a given temperature and moisture content) into the kinetic model to predict the starch gelatinization. This C_∞ shows dependence on temperature too. The facts above essentially pose a challenge to investigate the time and temperature dependence of gelatinization process. Besides, not only time and temperature mentioned above, but the starch samples employed and the process parameters could

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influence the progression and extent of gelatinization (Schirmer et al., 2015).

However, there is little literature available to explain the individual influence of time and temperature on different gelatinization stages. On the one hand, a well-defined starch sample is needed, which could be controlled to a specific stage of gelatinization to measure its time dependence under isothermal conditions (Yamamoto et al., 2006). But it is difficult to divide the gelatinization process into distinct sequential stages since the boundary of the amorphous and crystalline regions of native starch is not clearly demarcated. Heat-moisture treatment (HMT) is a physical method applied to improve the thermal stability of starch granules by inducing molecular reorganization (Wang et al., 2016; Xing et al., 2017c). In our previous study (Xing et al., 2017b), a multi-stage gelatinization theory was suggested in terms of heat-moisture treated starch through the comparative analysis of rheological and thermal properties during the same gelatinization process. And the gelatinization of starch could be well defined as three consecutive stages, ie. A, B, and C stage (Xing et al., 2017b), which makes it possible to use HMT starch as the test samples to study the time-temperature dependence of gelatinization process at different stages (Ratnayake and Jackson, 2006). In this study, the heat-moisture treated starch will be used as the test specimen.

On the other hand, both temperature and time change simultaneously during the heating-up process of starch. This makes it difficult to quantify and explain the independent effects of time and temperature on starch gelatinization at a constant heating rate (Schirmer et al., 2015). Therefore, a new experiment protocol needs to be developed to investigate the individual time and temperature dependence of starch gelatinization (Schirmer et al., 2015). The rheological properties of starch dispersions including the apparent viscosity, shear thinning and thickening behaviors can report important information on the starch gelatinization process (Ai and Jane, 2015; Moreira et al., 2012). This rheological study is also a good choice to model the gelatinization with different temperatures and shear rates (Lagarrigue and Alvarez, 2001). From the rheological viewpoint based on the flow properties of starch gelatinization, a new time-temperature gradient gelatinization (TTGG) method with a short (5 min) and two long (20 and 30 min) holding periods at a range of temperatures is developed in this study. This isothermal holding gelatinization method is similar to the one used earlier by Pérez-Santos et al. (2016). The variation of viscosity will be examined as an indicator to study the time and temperature dependent behaviors.

The main objective of this work is to investigate the time and temperature dependence of starch gelatinization process. Also, threshold temperatures or critical temperatures at which different stages of gelatinization occur will be also determined and quantified. Therefore, further insights gained in this study on the time-temperature dependence of the gelatinization process of starch will help starch-based industries to make better use this important natural resource.

2. Materials and methods

2.1. Preparation of heat moisture treated starch

Corn starch was obtained from Hebei Zhangjiakou Yujing Food Co., Ltd. (Hebei, China). The native corn starch was subjected to heat-moisture treatment according to our previous works with slight modifications (Xing et al., 2017a, b). The moisture content of starch samples was firstly measured and then adjusted to 30% (w/w) by spraying calculated amount of distilled water. These starch-water mixtures were sealed and equilibrated in Duran glass containers for 24 h at ambient temperature, then were placed in a forced air oven maintained at 120 °C for 30 min. After the heat-moisture treatment, the samples were air dried at 40 °C, grounded, and packed in zipper bags for further study.

2.2. Methods

2.2.1. Rheological tests

To understand the time and temperature dependent behaviors of the gelatinization, steady shear tests were performed on HMT starch samples using AR2000ex Rheometer (TA Instruments Ltd., New Castle, USA) with aluminum parallel plate geometry (40 mm diameter, 1 mm gap). In these tests, 10% (w/w) starch-water slurry samples were prepared by suspending HMT starch in distilled water. Rheometer was operated at a constant shear rate of 300 s^{-1}. All rheological tests were performed in triplicate and the data were average. A thin layer of silicone oil was applied between the clamp and the sample plate in order to prevent the water evaporation. An equilibration time of 2 min was maintained before each measurement.

2.2.2. Heating-rate dependent gelatinization tests

Temperature sweep tests were performed on starch slurry samples at four heating rates of 5, 10, 15, 20 °C/min (Fig. 1). The apparent viscosity was measured during these temperature sweep tests when the samples were heated from 25 to 95 °C at these heating rates. The variation of viscosity against time (in 1 s interval) was recorded.

2.2.3. Time-temperature gradient gelatinization (TTGG) tests

To study the time-temperature dependence at different stages of starch gelatinization, the time-temperature gradient gelatinization (TTGG) tests were carried out. TTGG involved a programmed step-wise temperature increase at a certain heating rate. Here, the apparent viscosity of starch-water mixture was monitored in real time (in 1 s interval) when the starch suspension was heated at a constant rate (10 °C/min) to a predetermined temperature, held at that temperature for different lengths of time and then further heated a final temperature (95 °C) at the same heating rate (Fig. 2). A short holding time of 5 min was firstly implemented. The temperature gradients, i.e. the temperatures at which the samples were held for a predetermined time, are from 64 to 95 °C (64, 66, 67, 68, 70, 71, 72, 73, 74, 76, 78, 80, 82, 84, 86, 88, 90, and 95 °C). Among which, several gradient temperatures (70, 71, 72, 74, 76, 78, 80, and 86 °C) were then specially selected for long holding times of 20 and 30 min. A constant heating rate of 10 °C/min was used in these TTGG tests. The viscosity data were plotted using both semi-logarithmic and linear coordinates for better illustration of the gelatinization behaviors. The viscosity difference before and after the heat-holding (5, 20, 30 min) were recorded and compared.

2.3. Time/temperature dependence

The $\Delta\mu$ value is the difference in viscosity at the beginning and end of the constant temperature holding. As stated above, there were short (5 min) and long (20 and 30 min) holding time. Based on these ramping and holding protocols, the time and temperature dependent behavior of starch gelatinization can be determined as follows.

(1) Temperature dependence (TEMD): The entire gelatinization during the heating process could be considered as temperature dependent since beginning of starch gelatinization. According to the statements presented by Wirakartakusumah (1981), the gelatinization stages are considered as temperature-dependent if the starch is not