

## Structural variation of rice starch in response to temperature during microwave heating before gelatinisation

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### ABSTRACT

Although it is well known that structural variations occur in starch during gelatinisation, little is known about how the structure of starch changes at subgelatinisation levels. The objective of this study was to investigate structural variations of rice starch ascribed to the temperature during microwave heating. Rapid conduction heating was used to imitate the high microwave heating rate through oil bath, which was then compared with traditional conduction heating. Structural changes due to temperature increases were investigated using thermogravimetry and differential scanning calorimetry while the distinct lamellar organisation of starch was obtained through small-angle X-ray scattering. The results showed that the structure of starch responds non-monotonically to temperature rising before gelatinisation, which was also affected by heating rates. The samples treated by microwave and rapid conduction heating essentially underwent the same thermal property changes and the molecular vibration of the microwaves did influence the submicroscopic lamellar structure.

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### 1. Introduction

Starch constitutes a major component of many foods. Changes in the structures of starch granules can lead to variations in physical properties on a macroscopic scale (e.g., rheology), which influence the quality and processing of starch-based food products (Aggarwal & Dollimore, 1998; Blazek & Gilbert, 2011; Colonna, Tayeb, & Mercier, 1989). Hence, studies that focus on the structural changes in starch granules during food processing are of great significance in improving and controlling the quality of food.

The structure of a native starch granule is defined in terms of amorphous and semi-crystalline growth rings (Vandeputte, Vermeylen, Geeroms, & Delcour, 2003). Linear amylose molecules and probably less ordered amylopectin are present in an amorphous state within each native granule (Morrison, 1995; Primo-Martin, Van Nieuwenhuijzen, Hamer, & Van Vliet, 2007). Every semi-crystalline growth ring is composed of repeats of alternating amorphous and crystalline lamellae (Barsby, Donald, & Frazier, 2001), which are related to branch points of the amylopectin side

chains and the short-chain fractions of amylopectin arranged as double helices and packed in small crystallites, respectively (Imberty, Buléon, Tran, & Pérez, 1991; Svensson & Eliasson, 1995; Tester, Karkalas, & Qi, 2004; Vandeputte et al., 2003). The semi-crystalline nature of starch is ascribed to the double helices formed by the hydrogen bond interactions of the amylopectin branches (Blazek & Gilbert, 2011; Fitzgerald, 2004; Hizukuri, 1986). Therefore, the strength of its hydrogen bonds largely determines the structure of a starch granule.

During the traditional processing of starch-based materials, starch granules are often treated using physical fields such as conduction heating or electromagnetic radiation. Hence, the interactions among hydrogen bonds within starch molecules are often affected through changes of orientation or intensity, resulting in changes in the semi-crystalline structure of starch. Studies have recently examined changes in the structure of starch granules in response to the relevant physical fields. Liu et al. (2009) reported that high-pressure processing at up to 10.0 MPa was not sufficient to destroy the crystalline structure of waxy corn starch molecules during gelatinisation process. Palav and Seetharaman (2007) found that the granules ruptured and there was a lack of swelling in starch at gelatinisation levels, when wheat starch dispersions were treated using microwave heating. However, little literature includes the studies investigating the response of hydrogen bond interactions to heating at subgelatinisation temperatures.

Abbreviations: MV, microwave heating; RCV, rapid conventional heating; CV, conventional heating.

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We focus on the effect of microwave heating on the starch during subgelatinisation, which is defined as the process in which the final temperature is lower than the temperature at which gelatinisation begins ( $T_0$ ) for raw rice starch measured by DSC. However, the effect of microwave heating on starch granules has typically either been related to the fast heating rate, as reported by Bilbao-Sáinz, Butler, Weaver, and Bent (2007), or to the strong vibrational motion of the polar molecules (Palav & Seetharaman, 2006). Therefore, in this study, an oil bath (RCV) was used to imitate the high heating rate seen in microwave heating, to develop a better understanding of structural changes in rice starch granules at subgelatinisation temperatures when treated with microwave heating (MV), rapid conventional heating (RCV) and conventional conduction heating (CV).

## 2. Materials and methods

### 2.1. Materials

Rice starch (protein content  $\leq 0.23\%$ , starch content  $\geq 94.22\%$ , amylose content of  $15.11\%$ , particle size of  $5.52 \pm 0.23 \mu\text{m}$ ) was isolated and purified from fresh rice (QiuShouBao Rice Products Co., Ltd., Anhui, China), as described in the previous works (Fan, Ma, Wang, Huang, et al., 2012; Fan, Ma, Wang, Zhao, et al., 2012).

### 2.2. Methods

#### 2.2.1. Heating conditions

**2.2.1.1. Rapid conventional heating (RCV).** A thermostat (Thermo Fisher Scientific Co., Ltd., MA, USA) with an oil bath ( $200^\circ\text{C}$ ) was used for rapid heating. The starch suspensions (100 g) with a concentration of  $6\%$  (w/w) were prepared with distilled water and heated to the desired temperature in the oil bath under constant agitation using an agitator (RW20, IKA Co., Ltd., Germany). The temperature was monitored online using an optical fibre probe (FISO Technologies Inc., Québec, Canada). The suspensions were then immediately placed into an ice bath to stop the gelatinisation process. The treated suspensions were freeze-dried, and then the starch samples were smashed in order to pass through a  $75\text{-}\mu\text{m}$  sieve.

**2.2.1.2. Microwave heating (MV).** The MV experiment was conducted in the same manner as that described above, except a microwave oven was used for heating. A 2450 MHz oven (Xianou Instrument Manufacturing Co., Ltd., Nanjing, China) was used to match the heating rate obtained in the conventional heating using an oil bath. Multiple replications were performed in the preliminary stages to determine the power necessary to match the oil bath.

**2.2.1.3. Conventional heating (CV).** The CV experiment was conducted in the same manner as that described above, except that the sample mass was increased to 200 g and a hot plate was used for heating. Its measured heating rate was  $0.13^\circ\text{C/s}$ .

#### 2.2.2. Thermal analyses

**2.2.2.1. Thermogravimetry.** The thermogravimetric analysis was performed with a Mettler–Toledo TGA/SDTA851E (Mettler–Toledo AG, Greifensee, Switzerland). The operation was conducted from  $30$  to  $700^\circ\text{C}$  at heating rates of 5, 10, 15 and  $20^\circ\text{C/min}$  under nitrogen, and a sample mass of 5 mg was maintained throughout the test. The thermogravimetric data were analysed according to BS ISO 11358-2 (British Standard Institution, 2005).

**2.2.2.2. Differential scanning calorimetry.** The starch sample heated to different final temperatures was analysed with a Q2000 differential scanning calorimeter (TA Instruments, New Castle, USA).

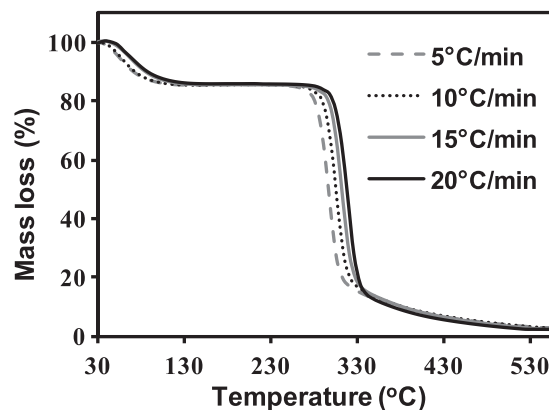


Fig. 1. Thermogravimetry curves of MV40 °C (starch heated by microwave to  $40^\circ\text{C}$ ) at heating rates of 5, 10, 15 and  $20^\circ\text{C/min}$ .

Approximately 8–10 mg of the freeze-dried sample was weighed into a standard aluminium sample pan. Each sample was scanned from  $30$  to  $90^\circ\text{C}$  at the rate of  $10^\circ\text{C/min}$ . A sealed empty pan was used as a reference.

#### 2.2.3. Small-angle X-ray scattering

SAXS experiments were performed on a SAXSess mc<sup>2</sup> nanostructure analyser (Anton Paar, Austria) with an incident X-ray monochromatic beam ( $\lambda_{\text{CuK}\alpha} = 1.54 \text{ \AA}$ ) monitored by a photomultiplier. The rotating anode device was operated at 40 kV and 50 mA. Scattering was detected in the range defined by the scattering vector  $q$  [ $q = 4 \sin(\theta)/\lambda$ , with  $\lambda$  being the wavelength and  $\theta$  the scattering angle]  $0.04\text{--}2.70 \text{ nm}^{-1}$ . Transmission, dark current and aluminium foil corrections were performed on the 2D images before further data processing. The data were analysed after desmearing with SAXSQuant software (Anton Paar, Austria). Dried starch samples were weighed ( $8.0 \pm 0.3 \text{ mg}$ ) and placed on the aluminium foils. Water was carefully added ( $8 \mu\text{L}$ ) to the starch and the samples were sealed to prevent any change in the amount of water present during the experiments (Cardoso & Westfahl, 2010). Measurements were performed at room temperature and each SAXS pattern was cumulated for 7 min.

All analyses were conducted in triplicate and the average values were reported.

## 3. Results and discussion

### 3.1. Comparison of heating rates between MV and RCV

Our previous study focusing on the gelatinisation of rice starch heated by MV compared the time–temperature profiles for MV and RCV below  $85^\circ\text{C}$  (Fan, Ma, Wang, Huang, et al., 2012; Fan, Ma, Wang, Zhao, et al., 2012). In those results, above  $70^\circ\text{C}$ , the heating rate of RCV decreased, while that of MV increased. Hence, we had to use the multi-stage MV heating program to match the profile of RCV. However, the highest heating temperature in this study was  $70^\circ\text{C}$ , therefore only one microwave power (1.2 kW) was used here. The time–temperature curves for MV and RCV completely coincided, and the heating rate of both MV and RCV was  $1.12^\circ\text{C/s}$  (the figure of temperature curves was not shown).

### 3.2. Thermal property analysis

#### 3.2.1. Thermogravimetry (TG)

The activation energy ( $E_a$ ) analysed from TG thermograms (Fig. 1) was used to describe the thermal stability caused by different heating methods (Guinesi et al., 2006; Marques et al., 2006). The

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