******Macintosh HD:Users:Peter:Desktop:OAlogo.jpg

***logo%20sjfs.wmf***

***Potravinarstvo Slovak Journal of Food Sciences***

**vol. 15, 2021, p. 824-834**

**https://doi.org/10.5219/1647**

Received: 6 June 2021. Accepted: 7 September 2021.

Available online: 28 September 2021 at www.potravinarstvo.com

© 2021 *Potravinarstvo Slovak Journal of Food Sciences*, License: CC BY 4.0

ISSN 1337-0960 (online)

THE EFFECT OF HEAT-MOISTURE TREATMENT CONDITIONS ON THE STRUCTURE PROPERTIES AND FUNCTIONALITIES OF POTATO STARCH

Chunli Deng, Oksana Melnyk, Yanghe Luo

ABSTRACT

Potato starch was modified via heat-moisture treatment (HMT) under various reaction conditions. The effect of HMT on structural, physicochemical, and in vitro digestibility were investigated. HMT led to the rupture, adhesion and partial gelatinization, and agglomeration of the granules which surface became rougher, thereby increasing the particle size and resulting in the hollow structure located at the hilum of potato starch granules. XRD results showed an increased relative crystallinity and transformed crystalline structure from B-type to C-type with the extension of heat moisture treatment. FTIR spectroscopy results indicated that HMT might cause additional interactions between amylose-amylose, and/or amylose-amylopectin and/or amylopectin-amylopectin chains, which eventually leads to the increase of the mass of the carbonyl group and a hydroxyl group. HMT significantly decreased the peak viscosity, hold viscosity, and breakdown viscosity of starch, while the gelatinization temperature increased. The tHMT1, THMT100, and CHMT15 had the lowest content of RDS and there was no significant difference between the three samples, that is these three samples had the highest total content of SDS and RS.

Keywords: potato starch; heat moisture treatment; structures properties; physicochemical properties; in vitro digestibility

Introduction

Starch is an important plant raw material for food, medicine, and other industrial application due to its advantages of being renewable, nontoxic, biodegradable, and relatively low cost **(Thakur et al., 2019)**. Meanwhile, starch-based derivatives are nowadays used for many applications in food processing to achieve particular technological properties **(Lu et al., 2014)**. Consequently, to meet the requirements of food and other industrial production, various approaches have been applied to enhanced starch properties, including physical, chemical, and enzymatic modifications. Compared with chemical modification, physical modification is a more attractive method due to the absence of extra chemicals and derivative products in the modified starch, **(Xie et al., 2018)**.

Different starch modification techniques regarding properties and applications have been comprehensively discussed in earlier publications **(Li, 2018)**. Heat moisture treatment (HMT) is a physical treatment that heats the starch granules at the low moisture content (<35%) and a high temperature (80 – 140 ℃) being above the glass transition temperature, but below the gelatinization temperature **(Sui et al., 2015)**. HMT promotes changes in the structure of starch chains, resulting in granular swelling, thermal stability, and paste property changes **(Colussi et al., 2017)**.

Scientific hypothesis

Due to the diversity of reaction conditions, including the botanical source, moisture content, temperature, treatment time, heat source, and cooling progress, it is difficult to define the properties of HMT starch. Therefore, this project was carried out to evaluate the effects of moisture levels, heating time, and temperature on morphological, pasting behavior, and structural characteristics as well as vitro digestibility properties of potato starch. We are expecting to provide new knowledge on HMT affecting potato starch, we are also expecting to broaden HMT starch application in the food field.

MATERIAL AND METHODOLOGY

**Samples**

Native potato starch (NS) was obtained from an unknown cultivar of potatoes purchased from a local market with the technology of wet milling extraction in Hezhou, China.

**Chemicals**

Porcine pancreatic α-amylase (EC 3.2.1.1, Shanghai Yuanye Bio-Technology Co., Ltd, China).

Amyloglucosidase from Aspergillusniger (EC 3.2.1.3, Shanghai Yuanye Bio-Technology Co., Ltd, China).

**Instruments**

Electric thermostatic drying oven (DH411C, Yamto, Japan).

Laser diffraction particle size analyzer (BT-2001, Baxter Instruments Co. LTD, China).

Scanning electron microscopy (JSM-7610F, JEOL, Japan).

X-ray diffractometer (Rigaku Ultima IV, Ultima IVTM, Japan).

FT-IR spectrophotometer (Spectrum 400, Perkin Elmer, USA).

Rapid viscosity analyzer (RVA-Starch Master2, Perten Instruments, Sweden).

**Laboratory Methods**

### Particle size determination

The particle size parameters of starch samples were detected by a laser diffraction particle size analyzer (BT-2001, Baxter Instruments Co. LTD, China) with a dry method. Air was used as a medium and the optical mode was Mie. A starch sample (3 g) was added to the storage hopper with a shading ratio ranging from 5% – 12%.

### Methods of scanning electron microscopy

The morphology of starch granule was observed by scanning electron microscopy (JSM-7610F, JEOL, Japan) as **Li (2018)** method with slight modification. The starch samples were sprinkled on a double-sided adhesive tape mounted on the sample stage made of aluminum and coated with gold with for 30 s by using an EDT-2000 ion sputter (2×10 – 4 MPa, 25 mA). Subsequently, all the coated samples were examined at an acceleration voltage of 2.0 kV and the images were captured at ×1500 magnification.

### Methods of X-ray diffraction (XRD)

An X-ray diffractometer (Rigaku Ultima IV, Ultima IVTM, Japan) with Cu-Kα radiation was used to evaluate the crystalline patterns of starch samples. The measurement was operated at 40 kV and 40 mA with the scanning region of the diffraction ranged between 4° and 40° with a step length of 0.02° and the scan speed was 4°/min **(Zhao et al., 2019)**. The relative crystallinity was calculated by the ratio of the relative area of crystalline peaks to the total area using MDI Jade 6 software as follows:

### Methods of Fourier transform infrared spectroscopy (FT-IR)

The Fourier transform infrared spectra of starch samples were recorded on an FT-IR spectrophotometer (Spectrum, Perkin Elmer, USA) with a scanning spectral range from 4000 cm-1 to 400 cm-1 at 25 °C temperature. The starch powder was placed on to sampling unit and then to be measured directly **(NidhiDangi, Baljeet and Ritika, 2019)**.

### Analysis of the starch viscosity properties

The starch viscosity properties of starch samples were evaluated using an RVA Starch Master 2 (Perten Instruments, Sweden) with standard RVATM potato starch methodology. A 2.5 g starch sample (corrected to 14% moisture content) was dispersed in 25 mL distilled water in the aluminum RVA canister. The starch suspensions were heated to 50 ℃ in 60 s, heating from 50 ℃ to 95 ℃ in   
222 s and maintained at 95 ℃ for 150 s, and then were cooled to 50 ℃ in 228 s, and maintained at 50 ℃ for 108 s. A whole RVA test needed 780 s. The rotational speed was 960 rpm for the first 10 s and followed rotation speed was 160 rpm. The parameters, including gelatinization temperature, peak viscosity, breakdown, setback, and final viscosity were determined.

### Analysis of in vitro digestibility

The rate of in vitro digestion of native starch (NS) and HMT starch was determined using the Englyst method following the method of **Xie et al. (2018)**. Each Starch sample (200 mg dry basis) was suspended in a   
100 mL centrifuge tube containing 10 mL of sodium acetate buffer solution (0.1 M, pH 5.2) by vortexing for 1 min. And then the centrifuge tube was cooked in boiling water for   
30 min, then was removed and cooled to 37 ℃. Another   
10 mL of sodium acetate buffer solution (0.1 M, pH 5.2) was used as a blank control. Then five glass balls were added to the sample centrifuge tube, and all the experimental samples and blank control samples were incubated at 37 ℃ for 10 min with shaking (160 rpm). Then 5 mL α-glucoamylase enzyme solution (300 U.mL-1) and   
2 mL amyloglucosidase enzyme solution (225 U.mL-1) were added to the sample centrifuge tube, followed by incubation in a water bath at 37 °C with shaking (190 rpm). Aliquots (1 mL) of the hydrolyzed solution were taken at 20 and   
120 min and mixed with 20 mL of ethyl alcohol to deactivate the enzymes. The mixed solution was centrifuged at 4000 rpm for 10 min, and the glucose content in the supernatant was investigated using the GOPOD assay kit (Megazyme, Co., Wicklow, Ireland). Based on the hydrolysis rate, starch was defined following digestion speed as follows: rapidly digested starch (RDS, digested within 20 min), slowly digested starch (SDS, digested between 20 and 120 min), and resistant starch (RS, residual starch after 120 min), respectively. The RDS, SDS, and RS contents were calculated following the following respective formulas:

(2)

(3)

(4)

Where:

G20 is the glucose content released at 20 min; G120 is the glucose content released at 120 min; FG is the glucose content in the sample before enzymatic treatment (in terms of 0) and TS is the mass of the total starch.

**Description of the Experiment**

**Sample preparation:** The moisture content of native starch powder (NS) was determined before heat- moisture treatment, and then heat-moisture treatment modified potato starch samples were prepared as follow: (1) Different heating time groups (tHMT): 70 g native starch powder was weighted in 500 mL Duran laboratory bottle, and ultrapure water was added to adjust moisture content to 25% and equilibrated for 24 hours at 25 ℃, samples were then heated at 110 ℃ for 1 h, 2 h, 3 h, 4 h,5 h and 6 h ( tHMT1, tHMT2, tHMT3, tHMT4, tHMT5, tHMT6, respectively ) in a hot-air oven. (2) Different heating temperature groups (THMT): Starches were weighted in different spiral blue-moth bottles, and ultrapure water was added to adjust moisture content to 25% and equilibrated for 24 hours at 25 ℃, samples were then heated at 90 ℃, 100 ℃, 110 ℃, 120 ℃, 130 ℃ (THMT90, THMT100, THMT110, THMT120, THMT130, respectively) for 2 h in a hot- air oven. (3) Different moisture content of starch system groups (CHMT): Starches were weighted in different spiral blue-moth bottle, and different volumes of ultrapure water were added to adjust moisture content to 15%, 20%, 25%, 30%, 35%（CHMT15, CHMT20, CHMT25, CHMT30, CHMT35, respectively）and equilibrated for 24 h at 25 ℃, samples were then heated in an electric thermostatic drying oven for 2 h. All these three groups of treated samples were dried in a drying oven at 45 °C for 24 h to make sure the moisture content of the treated samples was less than 12%. The dried potato starch was pulverized for 45 s using a universal pulverizer, passed through an 80-mesh sieve, vacuum-packed in polyethylene bags, and stored in an airtight container for conducting further studies.

Statistical Analysis

Results are presented as mean standard deviation (SDM). Statistical analysis was performed using the analysis of variance tool in the DPS (7.05) environment. The obtained data from the particle size determination, the analysis of the starch viscosity properties and in vitro digestibility were subjected to analysis of variance (ANOVA) and theDuncan Multiple Range Test (DMRT), and the values were considered significantly different when *p* ≤0.05.

RESULTS AND DISCUSSION

## Particle size analysis

The particle size distributions of heat moisture treatment starch and native potato starch granules were shown in Table 1 to Table 3. As can be seen from Table 1 to Table 3. The D10, D50, D(4,3), and D (3,2) of HTM starch were significantly higher than that of NS except for the 50 value and D(4,3) of tHMT6, whereas the S.S.A. value of HMT was significantly lower than NS. The temperature and moisture content had a great influence on the D90 value of HMT starch. The large particle size of HMT starch indicated that heat moisture treatment could result in the slight swell of potato starch, which might further lead to the rupture, adhesion and partial gelatinization, and agglomeration of the granules. This result was in agreement with a previous study about lily starch **(Li et al., 2020)**.

**Table 1** Effect of heat-moisture treatment on the particle size distribution of potato starch under a different holding time.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Samples** | **D10 (μm)** | **D50 (μm)** | **D90 (μm)** | **D(4,3) (μm)** | **D(3,2) (μm)** | **S.S.A(m2.kg-1)** |
| NS | 17.30 ±0.49d | 34.12 ±0.40c | 60.24 ±0.47ab | 36.60 ±0.38c | 22.24 ±0.56d | 99.93 ±2.54a |
| tHMT1 | 18.37 ±0.22c | 35.33 ±0.19b | 61.61 ±0.30ab | 37.83 ±0.18ab | 24.02 ±0.14c | 92.50 ±0.52b |
| tHMT2 | 18.67 ±0.03c | 35.30 ±0.14b | 61.51 ±0.59ab | 37.84 ±0.21ab | 24.27 ±0.06c | 91.52 ±0.20b |
| tHMT3 | 19.43 ±0.13ab | 35.23 ±0.94b | 58.49 ±4.10bc | 37.18 ±1.35bc | 25.90 ±1.25ab | 85.99 ±4.17cd |
| tHMT4 | 18.95 ±0.13bc | 36.16 ±0.122a | 63.28 ±0.44a | 38.75 ±0.12a | 25.00 ±0.21bc | 88.87 ±0.74bc |
| tHMT5 | 18.59 ±0.14c | 35.37 ±0.28b | 61.45 ±0.31ab | 37.85 ±0.27ab | 24.48 ±0.19c | 91.63 ±0.48b |
| tHMT6 | 19.80 ±0.41a | 34.73 ±0.46bc | 56.39 ±2.33c | 36.50 ±0.76c | 26.51 ±0.39a | 83.83 ±1.23d |

**Table 2** Effect of heat-moisture treatment on particle size distribution potato starch under different holding temperatures.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Samples** | **D10(μm)** | **D50(μm)** | **D90(μm)** | **D(4,3) (μm)** | **D(3,2) (μm)** | **S.S.A(m2.kg-1)** |
| NS | 17.30 ±0.49c | 34.12 ±0.40c | 60.24 ±0.47c | 36.60 ±0.38c | 22.24 ±0.56c | 99.93 ±2.54a |
| THMT90 | 18.68 ±0.29b | 35.59 ±0.30ab | 61.78 ±0.33b | 38.06 ±0.27ab | 24.02 ±0.67b | 92.54 ±2.66b |
| THMT100 | 18.62 ±0.21b | 35.60 ±0.39ab | 61.85 ±0.55b | 38.08 ±0.36ab | 24.30 ±0.26b | 91.43 ±0.97b |
| THMT110 | 18.67 ±0.03b | 35.30 ±0.14b | 61.51 ±0.59b | 37.84 ±0.21b | 24.27 ±0.06b | 91.52 ±0.20b |
| THMT120 | 19.36 ±0.42a | 35.49 ±0.35ab | 59.77 ±0.09c | 37.86 ±0.42b | 26.09 ±1.13a | 85.27 ±3.79c |
| THMT130 | 18.84 ±0.17b | 35.87 ±0.16a | 62.69 ±0.47a | 38.46 ±0.18a | 24.92 ±0.19b | 89.15 ±0.68b |

**Table 3** Effect of heat-moisture treatment on the particle size distribution of potato starch under different moisture content.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Samples** | **D10(μm)** | **D50(μm)** | **D90(μm)** | **D(4,3) (μm)** | **D(3,2) (μm)** | **S.S.A(m2/kg)** |
| NS | 17.30 ±0.49c | 34.12 ±0.40d | 60.24 ±0.47e | 36.60 ±0.38d | 22.24 ±0.56c | 99.93 ±2.54a |
| CHMT15 | 18.53 ±0.08b | 35.25 ±0.11c | 61.25 ±0.25de | 37.71 ±0.14c | 24.17 ±0.11b | 91.95 ±0.40b |
| CHMT20 | 18.93 ±0.16b | 35.72 ±0.03c | 62.38 ±0.29bc | 38.33 ±0.02bc | 24.67 ±0.29b | 90.04 ±1.06b |
| CHMT25 | 18.67 ±0.03b | 35.30 ±0.14c | 61.51 ±0.59cd | 37.84 ±0.21c | 24.27 ±0.06b | 91.52 ±0.20b |
| CHMT30 | 18.81 ±0.29b | 36.38 ±0.21b | 63.37 ±0.90b | 38.68 ±0.46b | 24.92 ±0.46b | 89.17 ±1.67b |
| CHMT35 | 20.05 ±0.47a | 41.12 ±0.42a | 78.39 ±0.62a | 45.60 ±0.50a | 27.00 ±0.81a | 82.33 ±2.45c |

Note: all values are the means of triplicate determinations ±*SD*. The means within the same column with different letters are significantly different (*p* <0.05); D(4,3), particle diameter of volume; D(3,2), particle diameter of surface, S.S.A., specific surface area; D10, D50, and D90 represent the corresponding particle size which is smaller than 10%, 50%, and 90% of the sample particles, respectively.

Morphological properties

The microscopic images of the NS and HTM starch samples obtained under SEM were shown in Figure 1. Almost all the NS starch showed elliptical-shaped granules and some round-shaped granules with a smooth surface.

After heat moisture treatment, potato starch granules were damaged and showed a negligible change in terms of morphology at low moisture levels (≤25%), which was in agreement with the results of HMT modified highland barley starch granules at low moisture levels **(Liu et al., 2018)**. Relatively high temperatures (e.g., 120 ℃ and   
130 ℃) and high moisture content (e.g., 30% and 35%) led to a coarser and rougher granule surface than that of NS. Similar results were obtained for HMT modified rice starch that the granule surface became slightly rougher than that of NS with the increasing agglomeration degree of the starch granules **(Wang et al., 2019)**.

银色的照片

中度可信度描述已自动生成

图片包含 图示

描述已自动生成

手机屏幕截图

低可信度描述已自动生成

**Figure 1** Morphology of the native potato starch and HMT starch.

## Light microscopy

Normal light microscopy (NLM) and polarized light microscopy (PLM) was used to obtain the microscopic images of NS and HMT modified starch (Figure 2 and Figure 3). Smooth surface and hilum structure were obtained from NS granules under normal light. After HMT modification, the hilum structure of HMT potato starch became hollow. The diameter and depth of the hollow region increased with increasing the length of heating, moisture content, and heating temperature. Similar results were obtained by **Li et al. (2020)**. The location of the hilum structure which was in the amorphous zone led to relative fragility. Furthermore, high temperature and certain moisture conditions during HMT caused partial swelling and disruption of starch granules, which was the reason that the hollow central region became large.

Visible black polarization cross-like “X” and “十” of NS starch granules were observed under polarized light. Maltese cross with the helium was biased toward the small end of the starch granules, and the deviation of large granular starch was higher. However, for HMT starch, the contour of the Maltese cross became gradually unclear and showed a large black area in the middle of the cross. The Maltese cross contour became unclear by short-time HMT modification and became obvious when the heating length was over 5 h. the average orientation of helical structures caused birefringence patterns. The results were consistent with the results of previous research of K. Liu et al **(Liu et al., 2018)**. The decrease in birefringence intensity on HMT indicated a change in the radial orientation of double helices due to increased amylopectin chain flexibility.

## X-ray diffraction (XRD)

The diffraction patterns of NS and HMT starch samples were illustrated in Figure 4. Four main peaks at 5.6° 2θ, 17° 2θ, 22° 2θ and 24° 2θ can be seen from the NS starch pattern, indicating that the crystal form of potato starch was B-type. Some significant changes in the crystalline patterns of HMT starch were observed. With the extension of heat moisture treatment, all HMT starch diffraction peaks gradually disappeared at 5.6° 2θ and 19.5° 2θ, the diffraction peak at 15° 2θ became smooth, and the two independent diffraction peaks at 22° 2θ and 24° 2θ tended to merge into a single peak near 23° 2θ, which was a characteristic of A-type. When treatment time reached   
5 hours, the diffraction peak at 23° 2θ divided into two diffraction peaks at 22° 2θ and 24° 2θ again. These results indicated the crystal structure of potato starch gradually changed from B-type to C-type with the extension heat moisture treatment. When treatment time was too long (more than 5 hours), its crystal structure changed to B-type again.

Relative crystallinity for NS was 19.37% and the crystallinity of tHMT starch initially increased and then decreased with the extension of the treatment time, the tHMT3 sample had the highest crystallinity of 24.43% of tHMT group samples. As can be seen from Table 4 – Table 6, all HMT starch had higher crystallinity than that of NS. With the increase of treatment temperature from 90 ℃ to 130 ℃, the crystallinity increased from 21.30% to 25.74%. With the increase of moisture content from 15% to 35%, the crystallinity increased from 21.46% to 25.09%. The results indicated that heat moisture treatment destroyed and rearranged the double helix structure of starch molecules, which strengthened the connection between starch chains, produced a new and more stable crystalline structure, and ultimately led to an increasing crystallinity of starch. The increased X-ray intensity was agreed with previous studies about HMT mung bean starch **(Li, Ward and Gao, 2011)**.

## FTIR spectroscopy

The FTIR spectra of NS and HMT starches were showed in Figure 5. All samples showed the same characteristic peak at 3290, 2920, 1636, and 1344 cm-1 and no new absorption peaks were generated, which indicated that the primary structure of starch could not be changed by heat-moisture treatment and proved that heat-moisture treatment was a physical modification method. However, it can be seen from the spectrograms that within the spectrum range, the intensity of the absorption peaks was different with different modification conditions, although the position of absorption peaks did not change. The absorption peaks at 1600 cm-1 – 1750 cm-1 correspond to the carbonyl (>C=O) stretching vibration area **(Dankar et al., 2018)**. The absorption peaks at 2850cm-1 – 3100cm-1 correspond to the CH stretching vibration area and the absorption peaks at 3100cm-1 – 3700cm-1 corresponds to the hydroxyl (OH) stretching vibration area **(Kizil, Irudayaraj and Seetharaman, 2002)**. The absorption peak strength of potato starch increased or decreased, indicating that the internal arrangement and the secondary structure of starch molecular conformation changed in the process of heat-moisture treatment, moreover, HMT might cause additional interactions between amylose-amylose, and/or amylose-amylopectin and/or amylopectin-amylopectin chains, which eventually leads to the increase of the amounts of the carbonyl group and hydroxyl group.

## The starch viscosity properties

The starch viscosity properties are correlated to the gel texture properties, the stability of starch paste, and retrogradation tendency. The RVA profiles of NS and HMT starch were shown in Figure 6 and the gelatinization parameter values were shown in Table 5, Table 6, and Table 7. As shown in Fig.6, the RVA profiles of all HMT samples had a great difference with NS starch, the RVA gelatinization properties of HMT starches were significantly different from each other.

Compared with native potato starch, the peak viscosity, hold viscosity, and breakdown viscosity of HMT starch decreased with the increasing of moisture content, treatment temperature, and treat time, while the gelatinization temperature increased (Table 5, Table 6, and Table 7). It also can be seen from Table 5, Table 6, and Table 7, the final viscosity of HMT starch was higher than peak viscosity, which indicated that potato starch modified by heat-moisture treatment was more prone to retrogradation. The decrease of peak viscosity is supposedly attributed to the strengthening of starch interior interactions by heat moisture treatment enhancing granule integrity, preventing the penetration of water into the starch granules.

许多照片放在一起

描述已自动生成

一群不同颜色的方块

中度可信度描述已自动生成

不同颜色的方块

低可信度描述已自动生成

**Figure 2** Morphological characteristics of the heat-moisture treated potato starch granules under normal light × 40 (A).

图片包含 图示

描述已自动生成

图片包含 图示

描述已自动生成

图片包含 图示

描述已自动生成

**Figure 3** Morphological characteristics of the heat-moisture treated potato starch granules polarized light × 40 (B).

图表

描述已自动生成图表

描述已自动生成

图表

描述已自动生成

**Figure 4** X-ray diffractograms of native and HMT potato starch.

图表

描述已自动生成图表

描述已自动生成

图表

描述已自动生成

**Figure 5** FTIR spectra of native and HMT potato starch.

The breakdown viscosity is related to the thermal stability of the swollen starch granules in the starch paste during heating and shearing, i.e. lower breakdown indicates more resistance to shear force **(****Zhu, Mojel and Li, 2018;** **Karim et al.,2008)**.

The lower viscosity and higher gelatinization temperature indicated that the structure of starch particles was more stable and the internal molecule force was stronger after heat moisture treatment. Therefore, the starch modified by heat moisture treatment requires more energy to decompose its structure and form paste, thus improving the gelatinization of the sample. The setback viscosity decreased with the extension of treatment time, temperature, and moisture content of the HMT process. The setback viscosity has great relativity to the retrogradation properties of starch and has a great influence on the tensile strength of vermicelli food **(Liao and Wu, 2016)**.

**Table 4** Effect of heat-moisture treatment on rheological properties and relative crystallinity of potato starch under a different holding time.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Samples** | **Pasting temperature /℃** | **Peak viscosity /cP** | **Hold viscosity /cP** | **Final viscosity /cP** | **Breakdown /cP** | **Setback /cP** | **Relative crystallinity (%)** |
| NS | 68.53±0.23d | 6598.00±72.75a | 4071.67±25.40a | 4567.33±36.95a | 2526.33±47.34a | 495.67±11.55c | 19.37 |
| tHMT1 | 76.10±1.31c | 2189.00±63.24b | 2145.00±60.90b | 4234.00±71.14b | 44.00±2.65b | 2089.00±25.06a | 20.08 |
| tHMT2 | 76.73±1.62c | 1888.00±14.00c | 1850.00±10.15c | 3736.33±8.62c | 38.00±4.36bc | 1886.33±3.51b | 23.32 |
| tHMT3 | 95.03±0.06a | 173.67±4.51d | 148.67±4.04e | 573.00±9.54d | 25.00±1.00bc | 424.33±5.69d | 24.43 |
| tHMT4 | 97.00±0.00a | 62.67±4.93e | 232.33±22.12d | 232.33±22.12e | 6.67±0.58c | 176.33±17.50e | 22.83 |
| tHMT5 | 95.05±0.07a | 47.33±5.51e | 42.67±3.21f | 145.67±34.43f | 4.67±2.89c | 103.00±31.23f | 20.98 |
| tHMT6 | 90.93±3.97b | 42.33±1.53e | 38.67±0.58f | 109.33±2.08f | 3.67±1.15c | 70.67±2.31g | 20.75 |

**Table 5** Effect of heat-moisture treatment on rheological properties and relative crystallinity of potato starch under different holding temperatures.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Samples** | **Pasting temperature /℃** | **Peak viscosity /cP** | **Hold viscosity /cP** | **Final viscosity /cP** | **Breakdown /cP** | **Setback /cP** | **Relative crystallinity (%)** |
| NS | 68.53±0.23d | 6598.00±72.75a | 4071.67±25.40a | 4567.33±36.95b | 2526.33±47.34a | 495.67±11.55f | 19.37 |
| THMT90 | 72.73±1.99c | 3366.67±56.31b | 3299.33±53.29b | 6996.67±80.70a | 67.33±3.05b | 3697.33±71.14a | 21.30 |
| THMT100 | 67.73±3.00d | 1662.00±17.09d | 1617.33±15.04d | 3023.67±13.32d | 44.67±2.08bc | 1406±12.50c | 21.43 |
| THMT110 | 76.73±1.62b | 1888.00±14.00c | 1850.00±10.15c | 3736.33±8.62c | 38.00±4.36bc | 1886.33±3.51b | 23.32 |
| THMT120 | 74.97±1.03bc | 1281.67±35.25e | 1258.00±37.59e | 2364.67±30.43e | 23.67±5.51c | 1106.67±12.10d | 25.77 |
| THMT130 | 81.2±1.61a | 1165.00±25.06f | 1147.00±23.64f | 2001.67±59.21f | 18.00±1.73c | 854.67±35.91e | 25.74 |

**Table 6** Effect of heat-moisture treatment on rheological properties and relative crystallinity of potato starch under different moisture content.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Samples** | **Pasting temperature /℃** | **Peak viscosity /cP** | **Hold viscosity /cP** | **Final viscosity /cP** | **Breakdown /cP** | **Setback /cP** | **Relative crystallinity (%)** |
| NS | 68.53±0.23c | 6598.00±72.75a | 4071.67±25.40b | 4567.33±36.95b | 2526.33±47.34a | 495.67±11.55e | 19.37 |
| CHMT15 | 67.80±0.26c | 5594.33±31.26b | 4919.67±48.75a | 5929.67±73.21a | 674.67±41.88b | 1010.00±29.72d | 21.46 |
| CHMT20 | 66.33±3.89c | 1720.33±12.70d | 1685.33±14.47d | 3101.67±34.02d | 35.00±2.00c | 1416.33±26.10b | 21.71 |
| CHMT25 | 76.73±1.62b | 1888.00±14.00c | 1850.00±10.15c | 3736.33±8.62c | 38.00±4.36c | 1886.33±3.51a | 23.32 |
| CHMT30 | 83.80±0.17a | 1377.67±4.73e | 1345.67±7.57e | 2627.33±51.93e | 34.00±2.64c | 1275.00±36.35c | 24.19 |
| CHMT35 | 85.30±1.68a | 1217±9.54f | 1180.00±13.75f | 2190.33±26.58f | 37.00±5.29c | 1010.33±13.80d | 25.09 |

Note: all values are the means of triplicate determinations ±*SD*. The means within the same column with different letters are significantly different (*p* <0.05).

**Table** **7** RDS, SDS and RS contents of native and HMT starch.

|  |  |  |  |
| --- | --- | --- | --- |
| **Samples** | **RDS(%)** | **SDS(%)** | **RS(%)** |
| NS | 31.14±0.10a | 55.17±0.17a | 13.69±0.10h |
| tHMT1 | 28.28±0.30fg | 51.46±0.21fg | 20.25±0.10b |
| tHMT2 | 29.91±0.27c | 51.16±0.39g | 18.93±0.25de |
| tHMT3 | 30.46±0.71b | 50.28±0.26h | 19.25±0.16d |
| tHMT4 | 28.73±0.00ef | 49.11±0.16i | 22.16±0.16a |
| tHMT5 | 29.32±0.56d | 51.94±0.47f | 18.74±0.09e |
| tHMT6 | 29.93±0.10c | 50.05±0.17h | 20.02±0.09bc |
| THMT90 | 30.34±0.00bc | 52.59±0.48e | 17.07±0.48g |
| THMT100 | 28.38±0.18fg | 54.13±0.25b | 17.48±0.38f |
| THMT110 | 29.91±0.27c | 51.16±0.39g | 18.93±0.25de |
| THMT120 | 29.89±0.36c | 53.35±0.21cd | 16.76±0.16g |
| THMT130 | 30.30±0.17bc | 52.84±0.26de | 16.86±0.19g |
| CHMT15 | 28.20±0.27g | 53.93±0.18b | 17.88±0.09f |
| CHMT20 | 29.10±0.18de | 53.35±0.22cd | 17.54±0.09f |
| CHMT25 | 29.91±0.27c | 51.16±0.39g | 18.93±0.25de |
| CHMT30 | 28.78±0.50ef | 51.44±0.90fg | 19.79±0.41c |
| CHMT35 | 29.16±0.44de | 53.78±0.12bc | 17.06±0.35g |

Note: all values are the means of triplicate determinations ±*SD*. The means within the same column with different letters are significantly different (*p* <0.05).RDS :rapidly digested starch(digested within 20 min); SDS: slowly digested starch (digested between 20 and 120 min)；RS：resistant starch (residual starch after 120 min).

Therefore, it is necessary to select appropriate heat-moisture treatment conditions for the preparation of vermicelli food by using the HMT potato starch to obtain better edible quality.

## In vitro digestion of starch

The results of RDS, SDS, and RS of native starch and HMT starch sample were shown in Table 7. The values of RDS, SDS, and RS fractions in NS were 31.14%, 55.17%, and 13.69%, respectively. Compared to the native starch HMT starches had less RDS and SDS, but high RS. The total content of SDS and RS of HMT starch was significantly higher than that of NS starch (*p* <0.05), the holding time of heat-moisture treatment had the greatest effect on the content of RS, indicating that heat moisture treatment rearranged the molecular chain and formed ordered structures (crystallites and helices) and increased the resistance of starch to enzymatic hydrolysis, eventually reduce the digestion of starch by transforming of RDS into SDS and/or RS **(****Li, Ward and Gao, 2011)**. From our results, tHMT1, THMT100, and CHMT15 had the lowest content of RDS and there was no significant difference between the three samples, in other words, these three samples had the highest total content of SDS and RS. Therefore, choosing appropriate treatment time, temperature, and moisture content can obtain modified starch with higher content of SDS and RS.

CONCLUSION

HMT altered the particle size, morphological, structural, rheological, and digestive properties of potato starch at different levels of moisture content, heating time, and heating temperature. HMT led to large particle size, the rough surface of starch granules, and HMT resulted in the hollow structure located at the hilum of potato starch granules. XRD results showed an increased relative crystallinity in HMT starches and transformed crystalline structure from B-type to C-type. FTIR spectroscopy results indicated that the internal arrangement and the secondary structure of starch molecular conformation changed in the process of heat-moisture treatment, which eventually leads to the increase of the mass of the carbonyl group and hydroxyl group. In vitro digestion results showed that the RDS content of HMT starches significantly decreased, whereas the total content of SDS and RS increased, indicating HMT starches could potentially become sources of industrial-resistant starch and as low-calorie food ingredients.

referenceS

Colussi, R., Singh, J., Kaur, L., Zavareze, E. R., Dias, Á. R. G., Stewart, R. B., Singh, H. 2017. Microstructural characteristics and Gastro-small Intestinal Digestion In Vitro of Potato Starch: Effects of Refrigerated Storage and Reheating in Microwave. *Food Chemistry*, vol. 226, p. 171-178. <https://doi.org/10.1016/j.foodchem.2017.01.048>

Dankar, I., Haddarah, A., El Omar, F., Pujola, M., Sepulcre, F. 2018. Characterization of food additive-potato starch complexes by FTIR and X-ray diffraction. *Food Chemistry*, vol. 206, p. 7-12. <https://doi.org/10.1016/j.foodchem.2018.03.138>

Karim, A. A., Nadiha, M. Z., Chen, F. K., Phuah, Y. P., Chui, Y. M., Fazilah, A. 2008. Pasting and retrogradation properties of alkali-treated sago (Metroxylon sagu) starch. *Food Hydrocolloids*, vol. 22, p. 1044-1053. <https://doi.org/10.1016/j.foodhyd.2007.05.011>

Kizil, R., Irudayaraj, J., Seetharaman, K. 2002. Characterization of irradiated starches by using FT-Raman and FTIR spectroscopy. *J. Agric. Food Chem.*, vol. 50, no. 14, p. 3912-3918.

Li, G. 2018. Sweet potato starch modified by branching enzyme, b-amylase and transglucosidase. *Food Hydrocolloids*, vol. 83, p. 182-189. <https://doi.org/10.1016/j.foodhyd.2018.05.005>

Li, G., Jiahao, L., Hui, L., Yu, Z., Bo, C. 2020. The structure property and adsorption capacity of new enzyme-treated potato and sweet potato starches. *International Journal of Biological Macromolecules*, vol. 144, p. 863-873. <https://doi.org/10.1016/j.ijbiomac.2019.09.164>

Li, S., Ward, R., Gao, Q. 2011. Effect of heat-moisture treatment on the formation and physicochemical properties of RS from mung bean (*Phaseolus radiatus*) starch. *Food Hydrocolloids*, vol. 25, no. 7, p. 1702-1709. <https://doi.org/10.1016/j.foodhyd.2011.03.009>

Liao, L., Wu, W. 2016. Relationship between gelatinization and gel properties of different starch and their noodles. *Transaction of the Chinese Society of Agricultural Engineering*, vol. 30, no. 15, p. 332-338. <https://doi.org/10.3969/j.issn.1002-6819.2014.15.042>

Liu, D., Traverse, C. J., Chen, P., Elinski, M., Yang, C., Wang, L., Young, M., Lunt, R. R. 2018. Aqueous‐Containing Precursor Solutions for Efficient Perovskite Solar Cells. *Adv. Sci. (Weinh)*, vol. 5, no. 1, p. 1700484. <https://doi.org/10.1002/advs.201700484>

Lu, Y., Luthria, D., Fuerst, E. P., Kiszonas, A. M., Yu, L., Morris, C. F. 2014. Effect of Processing on Phenolic Compostion of Dough and Bread Fractions Made from Refined and Whole Wheat Flour of Three Wheat Varieties. *Journal of Agricultural and Food Chemistry*, vol. 62, no. 43, p. 10431-10436. <https://doi.org/10.1021/jf501941r>

NidhiDangi, Baljeet S. Y, Ritika, B. Y. 2019. Pasting, rheological, thermal and gel textural properties of pearl millet starch as modified by guar gum and its acid hydrolysate. *International Journal of Biological Macromolecules*, vol. 139, p. 387-396. <https://doi.org/10.1016/j.ijbiomac.2019.08.012>

Sui, Z., Yao, T., Zhao, Y., Ye, X., Kong, X., Ai, L. 2015. Effects of heat-moisture treatment reaction conditions on the physicochemical and structural properties of maize starch: Moisture and length of heating. *Food Chemistry*, vol. 173, p. 1125-1132. <https://doi.org/10.1016/j.foodchem.2014.11.021>

Thakur, R., Pristijono, P., Scarlett, C. J., Bowyer, M., Singh, S., Vuong, Q. V. 2019. Starch-based films: Major factors affecting their properties. *International Journal of Biological Macromolecules*, vol. 132, p. 1079-1089. <https://doi.org/10.1016/j.ijbiomac.2019.03.190>

Wang, Y., Wang, L., Zhou, X., Hu, S., Chen, H., Xiang, J., Zhang, Y., Zeng, Y., Shi, Q., Zhu, D., Zhang, Y. 2019. Research Progress on Heat Stress of Rice at Flowering Stage. *Rice Science*, vol. 26, no. 1, p. 1-10. <https://doi.org/10.1016/j.rsci.2018.06.009>

Xie, H., Gao, J., Xiong, X., Gao, Q. 2018. Effect of heat-moisture treatment on the physicochemical properties and in vitro digestibility of the starch-guar complex of maize starch with varying amylose content. *Food Hydrocolloids*, vol. 83, p. 213-221. <https://doi.org/10.1016/j.foodhyd.2018.04.038>

Zhao, S.-Y., Chen, Z.-Y., Wei, N., Liu, L., Han, Z.-B. 2019. Highly Efficient Cooperative Catalysis of Single-Site Lewis Acid and Brønsted Acid in a Metal–Organic Framework for the Biginelli Reaction. Inorg. *Chem*., vol. 58, no. 12, p. 7657-7661. <https://doi.org/10.1021/acs.inorgchem.9b00816>

Funds:

This research received no external funding.

Acknowledgments:

The authors gratefully acknowledge the financial support received from the Middle-aged and Young Teachers’ Basic Ability Promotion Project of Guangxi (Grant No. 2021KY0710) and the National Key R&D Program of China (Grant No. 2018YFD0901003).

Conflict of Interest:

The authors declare no conflict of interest.

**Ethical Statement:**

This article does not contain any studies that would require an ethical statement.

Contact Address:

Chunli Deng, Sumy National Agrarian University, Department of Food Technology, Herasyma Kondratieva str., 160, Sumy 40021, Ukraine; College of Food and Biological Engineering Hezhou University No.18, Xihuan Road, Hezhou 542899, Guangxi, P.R.China, Contact tel.:(0086)18277445653,

E-mail: [chunlideng2016@hzxy.edu.cn](mailto:chunlideng2016@hzxy.edu.cn)

ORCID: <https://orcid.org/0000-0003-1116-7407>

\*Oksana Melnyk, Sumy National Agrarian University, Department of Food Technology, Herasyma Kondratieva str., 160, Sumy 40021, Ukraine, Contact tel.: +38-096-432-8072,

E-mail: [oxana7@i.ua](mailto:oxana7@i.ua)

ORCID: <https://orcid.org/0000-0002-9201-7955>

\*Yanghe Luo, Institute of Food Science and Engineering Technology, Hezhou University, No.18, Xihuan Road, Hezhou 542899, Guangxi, P.R.China, Contact tel.:(0086) 774-5228635,

E-mail: [luoyanghe@tsinghua.org.cn](mailto:luoyanghe@tsinghua.org.cn)

ORCID: <https://orcid.org/0000-0003-2705-1505>

Corresponding author: \*