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Effect of heat-moisture treatment on physicochemical properties and digestive characteristics of sweet potato flour

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Abstract

In the present study, the effects of heat-moisture treatment (HMT) on the structural properties, physicochemical properties and in vitro digestibility of sweet potato flour were investigated. Moisture mass fractions of samples were set as 20%, 25%, 30% or 35%, and treated at 110 °C for 4 h. Gelatinization characteristics, short-range order, double helix structure, crystallinity and digestive characteristics were studied by test of pasting and thermal, analysis of X-ray diffraction spectra, infrared spectra and in vitro digestion test. The results showed that the structural characteristics of the sweet potato flour with different moisture concentrations were quite different. The viscosity, breakdown value and setback value of HMT-sweet potato flour were all lower than those of the original flour. The content of rapidly digestible starch (RDS) decreased, while the contents of slowly digestible starch (SDS) and resistant starch (RS) increased after HMT. The results showed that HMT-25 had good cold and heat stability, low pasting viscosity and high contents of SDS and RS.

Keywords: heat-moisture treatment; sweet potato flour; crystalline characteristics; pasting properties; double helix structure; in vitro digestibility.

Practical Application: Modification in physicochemical properties of sweet potato flour.

1 Introduction

Starch is a kind of high polymer compound, which is one of the main sources of human energy, so it is widely applied in food processing. The Amylose/amylopectin ratio in starch granules and the these two biopolymers are organized on different scales determine the structural properties of starch granules (Wang et al., 2016; Moloto et al., 2021). The microstructural properties of starch also determine its digestive properties in human body. Traditional sweet potato starch foods have a fast digestive rate in the body, leading to a rapid rise in human glycemic index, which has certain risks for special populations such as high blood sugar and high blood lipids. Therefore, it has great significance to explore how to prepare special foods with low digestion rate. Heat-moisture treatment (HMT) has been widely applied in starch modification due to its characteristics of environmental protection, safety and low cost (Hoover & Vasanthan, 1994). After modification, the structure of starch changes, the digestion rate in vivo decreases, and the blood glucose is kept stable (Wang et al., 2016; Li et al., 2017b). However, in the pasting process, the starch paste has some shortcomings such as poor shear resistance and fast regeneration rate, which leads to the product easy thinning, water separation and aging in the industrial production, resulting in poor product quality and short shelf life (Indrianti & Pranoto, 2018; Li et al., 2017b). Therefore, it has great significance to explore ways to change this situation.

Sweet potato powder as the research object in this experiment, the HMT was applied to the flour modified processing. Microstructure, gelatinization characteristics, short-range order, double helix structure, crystallinity and digestive characteristics were studied by observations on particle morphology, test of pasting and thermal, analysis of X-ray diffraction spectra, infrared spectra and in vitro digestion test. The HMT-sweet potato flour may not only have the characteristics of low digestibility and low viscosity is similar with HMT-sweet potato starch, but also have good cold and heat stability.

In the HMT modification technology, there were few reports about the HMT on sweet potato powder, but there were more reports about the entire flour rich in cellulose, ascorbic acid, sweet potato polysaccharide, minerals, phenolic substances and aldehydes and experiments in functional foods (Santos et al., 2019; Kourouma et al., 2020; Cartier et al., 2017; Franco et al., 2020; Dias et al., 2020). Therefore, HMT may not only improve the stability of the sweet potato flour products and maintain the low digestion characteristics of starch, but also the nutritional and functional characteristics of the sweet potato flour will have an important impact on human health. The test was carried out according to the test process of Scheme 1.

2 Materials and methods

2.1 Materials

sweet potato flour were provided by Henan Agricultural Research Institute; salt (Huai Brand) was bought from a local supermarket. alpha-amylase type VI-B from porcine pancreas (EC 3.2.1.1, A3176) was purchased from Sigma-Aldrich Chemical

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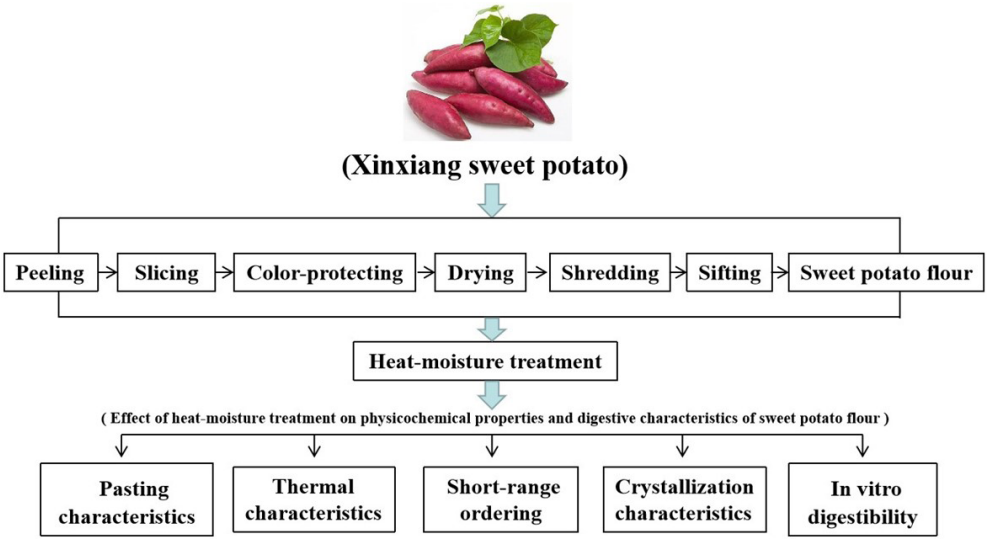

Scheme 1. Showing the experimental design of the study: 1) Xinxiang sweet potato was made into sweet potato flour through the processes of Peeling, slicing, color-protection, drying, shredding and sifting; 2) Heat- moisture modification treatment on sweet potato flour; 3) The pasting characteristics, thermal characteristics, short-range ordering, crystallization characteristics and in vitro digestibility characteristics of sweet potato flour after HMT were tested in order to explore the changes of its physicochemical properties and digestive characteristics.

Co. (St. Louis, MO, USA). Amyloglucosidase (EC 3.2.1.3) was purchased from Shanghai Yuanye Bio-Technology Co.,Ltd (Shanghai, China). All other chemical reagents were of analytical grade.

2.2 HMT of sweet potato flour

The method proposed by Wang et al. (2014) was adopted with some modifications. sweet potato flour (40 g) was accurately weighed into a screw-mouth bottle, and an appropriate amount of deionized water was added to adjust the moisture content to 20%, 25%, 30% and 35%. After stirring evenly, the bottle was sealed and kept at 4 °C to balance the moisture for 24 h. The screw-mouth bottle was then placed in a constant temperature blast drying oven, and the temperature was raised to 110 °C for 4 h. The HMT-sweet potato flour was dried at 40 °C for 24 h and then ground and screened through an 80 mesh sieve to obtain the finished product.

2.3 Determination of pasting characteristics

A 3 g sample of sweet potato flour and 25 mL of deionized water were accurately weighed, stirred evenly and placed in a special aluminum box for testing. The pasting characteristics were tested with a rapid viscosity analyzer (Super3, Newport Technology Corporation, Australia). The test procedure was run as follows: the sample was equilibrated at 50 °C for 2 min, heated at a rate of 6 °C/min to 95 °C, kept at 95 °C for 5 min, cooled at the same rate, and then kept at 50 °C for 2 min.

2.4 Determination of thermal characteristics

Thermal testing of the sample was carried out by differential scanning calorimetry (Q2000, TA Corporation, America). Five milligrams of sample and 15 μm of deionized water were accurately weighed and placed in a liquid crucible and then sealed and allowed to stand for 24 h at 4 °C to balance the moisture. The test procedure was set as follows: the temperature was raised at a rate of 10 °C/min, and the test temperature range was 30 °C to 130 °C.

2.5 Determination of infrared spectra

One milligram of sample and 100 mg of KBR were accurately weighed in an agate mortar for mixing grinding and tablet pressing (0.5 mm), and the sample was tested by Fourier transform infrared spectrometer (Thermo Electric Corporation, Waltham, MA, USA). The test parameters were set as follows: the scan range was 4 000 ~ 400 cm^{-1} and the resolution was 4 cm^{-1}. A DTGS detector was used, KBR was used as the background, and the infrared spectrum of the sample was obtained with 32 scans (Wang et al., 2014).

2.6 Determination of crystallization characteristics

The crystallinity of sweet potato flour samples was tested by X-ray diffractometer (D8 ADANCE, Bruker Corporation, Germany). The test parameters were designed as follows: 40 kV, 40 mA, the scan rate was 2°/min, and the diffraction angle (2θ) range was 3-30°.

2.7 Determination of flour digestion characteristics

A 200 mg sample was accurately weighed and placed in a test tube, 15 mL of 0.2 mol/L sodium acetate buffer (pH=5.2) was added, and then the tube was cooked in a boiling water bath for 10 min. When the sample cooled to room temperature, 10 mL of porcine pancreatic alpha-amylase (290 U/mL) and saccharase (15 U/mL) were added to the test tube, which was then placed in a 37 °C constant temperature water bath for shock digestion

(150 r/min) (Li et al., 2017a). After hydrolysis for 20 min and 120 min, 0.5 mL samples of hydrolysate were removed and put into centrifuge tubes, and 4 mL of anhydrous ethanol was added to inactivate the enzyme. After centrifugation (10 min, 6000 r/min), the supernatant was removed, and the absorbance value was determined by the DNS method at 540 nm. The mass fractions of rapidly digestible starch (RDS), slowly digestible starch (SDS) and resistant starch (RS) of the sample were calculated by the following Formulas 1, 2 and 3:

$$\mathbf{RDS}(\%) = (\mathbf{G_{20}} - \mathbf{FG}) \times \mathbf{0.9} / TS \times \mathbf{100}\% \quad (1)$$

$$\mathbf{SDS}(\%) = (\mathbf{G_{120}} - \mathbf{G_{20}}) \times \mathbf{0.9} / TS \times \mathbf{100}\% \quad (2)$$

$$\mathbf{RS}(\%) = \mathbf{TS} - (\mathbf{RDS} + \mathbf{SDS}) / TS \times \mathbf{100}\% \quad (3)$$

where G_{20} is the glucose content produced after amylase hydrolysis for 20 min (mg); FG is the free glucose content of starch before enzymatic hydrolysis (mg); G_{120} is the glucose content produced after amylase hydrolysis for 120 min (mg); and TS is the total starch content in the sample (mg).

2.8 Processing and analysis of data

Data were analysed by one-way analysis of variance (ANOVA), followed by Duncan's multiple range test using SPSS 17.0 Statistical Software Program (SPSS Incorporated, Chicago). A value of $p < 0.05$ was considered statistically significant. Results are expressed as the mean ± standard deviation of triplicate experiments.

3 Results and discussion

3.1 Pasting characteristics

The pasting of starch primarily reflects the characteristics of hot paste formation time, temperature, viscosity, shear resistance and recovery rate of the starch. Pasting data for the sweet potato flour were determined by rapid viscosity analyzer and are shown in Table 1. Peak viscosity, trough viscosity and final viscosity of HMT-sweet potato flour were lower than those of the original flour and showed a downward trend with increasing moisture content. This may be because starch particles swelled in water during HMT, hydrogen bonds broke, the double helix structure decomposed, long-range ordering and short-range ordering decreased, and the crystal structure was destroyed (Wang et al., 2014; Teba et al., 2009). These factors led to an increase in thermally induced dissolution and decreased cracking resistance of starch, easy pasting and decreased viscosity. It was also possible that the high temperature caused alteration and transformation of the starch grain crystal zone, an increase in the number of chains in the amorphous zone and strengthened interactions between the molecular chains of starch (Barrera et al., 2013). This phenomenon is similar to that seen in the research results of Marta et al. (2019) and Olayinka et al. (2008). It was also possible that protein and fat in the flour formed starch-fat and starch-protein complexes, which increased the rigidity of starch particles and enhanced swelling resistance (Chen et al., 2015). The breakdown value of starch indicates the resistance of starch hot paste to thermal shear force, which is related to the structural strength of starch granules. The setback value indicates the stability of starch hot paste, which is related to the content of amylose and the recombination orientation of amylopectin. As shown in Table 1, the breakdown value and setback value of HMT-sweet potato flour were both lower than those of the original flour. This may be because the interactions between amylopectins were strengthened during HMT, and the migration and flow of amorphous α-glucan promoted the rearrangement of the double helix structure and formed a tighter rigid structure (Suriya et al., 2019; Zhang et al., 2019). This indicates that the HMT-sweet potato flour has better shear resistance and stability. As shown in Table 1, among the pasting temperature parameters measured by RVA, only raw flour and HMT-20 showed pasting temperatures, and the pasting temperature of HMT-20 was significantly higher than that of raw flour, while the pasting temperatures of HMT-25, HMT-30 and HMT-35 might be so high that they could not be determined in RVA with the maximum temperature of 95 °C. This situation may also be one of the reasons why HMT-sweet potato flour was not completely gelatinized in RVA and its peak viscosity was lower than that of raw flour. The increase in the pasting temperature of HMT-sweet potato flour indicates that the starch molecular chains were rearranged and oriented, and the structural rigidity of the starch was strengthened after HMT (Cordeiro et al., 2018).

3.2 Thermal characteristics

As shown in Table 2, the T_o, T_p and T_c of HMT- sweet potato flour were all higher than those of raw flour. The reasons for this phenomenon may be related to the factors that caused the differences in pasting characteristics of HMT-sweet potato flour. The internal structure of sweet potato flour particles was greatly

Table 1. Pasting properties of raw and HMT-sweet potato flour.

Samples	Peak viscosity/ (mPa•s)	Trough viscosity/ (mPa•s)	Final viscosity/ (mPa•s)	Break down/ (mPa•s)	Set back/ (mPa•s)	peak time/ min	Pasting temperature/°C
RAW	1169.0 ± 34.7^a	652.3 ± 15.0^a	857.3 ± 21.9^a	516.7 ± 19.7^a	205.0 ± 7.0^a	4.2 ± 0.0^b	80.1 ± 0.1^b
HMT-20	397.3 ± 4.0^b	358.7 ± 3.5^b	516.0 ± 0.0^b	38.7 ± 0.6^b	157.3 ± 3.5^b	7.0 ± 0.0^a	93.7 ± 0.1^a
HMT-25	52.3 ± 1.2^c	48.7 ± 1.5^c	81.7 ± 4.0^c	3.7 ± 0.6^c	33.0 ± 2.6^c	6.9 ± 0.0^a	-
HMT-30	44.0 ± 2.0^c	38.7 ± 3.1^c	75.3 ± 4.2^c	5.3 ± 1.2^c	36.7 ± 1.2^d	6.9 ± 0.1^a	-
HMT-35	50.0 ± 1.0^c	44.0 ± 1.0^c	93.0 ± 2.0^c	6.0 ± 0.0^c	49.0 ± 1.0^d	7.0 ± 0.0^a	-

Values are means of three determinations (n = 3); values followed by different uppercase letters within a column differ significantly ($P < 0.05$).

Table 2. Thermal properties of raw and HMT- sweet potato flour.

Samples	T_o/°C	T_p/°C	T_c/°C	△H/(J/g)
RAW	72.76 ± 0.32^d	79.91 ± 0.15^e	84.72 ± 0.25^e	1.42 ± 0.02^e
HMT-20	84.63 ± 0.25^c	92.33 ± 0.37^c	104.25 ± 0.13^d	5.06 ± 0.03^a
HMT-25	84.83 ± 0.22^a	97.96 ± 0.7^a	114.49 ± 0.06^a	4.4 ± 0.25^b
HMT-30	84.70 ± 0.30^b	94.28 ± 0.09^b	108.30 ± 0.23^b	2.7 ± 0.31^c
HMT-35	84.70 ± 0.27^b	91.93 ± 0.31^d	107.26 ± 0.24^c	1.99 ± 0.02^d

T_o: initial temperature; T_p: peak temperature; T_c: termination temperature; ΔH: gelatinization enthalpy. Values are means of three determinations (n = 3); values followed by different uppercase letters within a column differ significantly ($P < 0.05$).

affected by HMT, which made the starch granules more rigid and more resistant to heat. On the one hand, this result may be caused by the reorientation and rearrangement of amylose and amylopectin as the original structures of starch particles were destroyed by HMT, resulting in a more ordered and compact molecular structure. The pasting of starch particles required added thermal energy, leading to an increase in gelatinization temperature (Shi et al., 2018). It was also possible that the activity of the starch molecular chain was increased in the system with higher moisture content, and the interactions between molecular chains were strengthened, resulting in a more compact internal structure (Chung et al., 2009). On the other hand, it may also be because there were certain quantities of protein and fat in the flour, which produced protein-starch and fat-starch complexes and new hydrogen bonds, which inhibited water absorption by particles and increased the stability of particles, leading to the rise in starch pasting temperature (Chen et al., 2015). It is also possible that some loose and easily gelatinized starch was gelatinized in the process of HMT, while the remaining starch produced a population of ungelatinized starch with a more perfect structure (double helix, crystal) under the action of HMT, and pasting of this fraction of starch required more heat (Liao et al., 2019). This was mutually verified by the phenomenon observed in the SEM figure mentioned above. △H is a reflection of the change in the double helix structure inside the starch granule (Wang et al., 2018). As shown in the table, the △H of HMT-flour was significantly higher than that of raw flour, and the increased △H indicates that HMT-flour has a better double helix structure, which is consistent with the results reported by Ahn et al. (2013) and Huang et al. (2016). △H gradually decreased with increasing moisture content of HMT and the gradual decreases of T_o, T_p and T_c at HMT30% and HMT35% may be due to the higher fluidity of starch chains inside starch granules in a high moisture environment, which made the structure loose (Liao et al., 2019). This may also be because the proportion of gelatinized starch increased with increasing HMT water content, and the proportion of starch with perfect rigid structures gradually decreased (Cordeiro et al., 2018).

3.3 Infrared spectra

Short-range ordering on the surface of starch particles is generally studied by Fourier-transform infrared spectroscopy because the infrared spectrum has a small detection distance of only 2 μm, which better reflects the amyloid chain structures on the surfaces of starch particles (Sevenou et al., 2002). Starch is a polycrystalline polymer with a specific crystal absorption band in the infrared spectrum that reflects its crystalline state (Wang et al., 2017). The higher the absorption band intensity is the higher the crystallinity (Wang et al., 2017). In the experiment, according to the method of van Soest et al. (1995), the absorption peaks intensity ratios 1047 cm^{-1}/1022 cm^{-1} and 1022 cm^{-1}/998 cm^{-1} were used to determine short-range ordering and the change in the double helix structure in the HMT-flour crystalline region. Figure 1 shows the infrared spectra of raw sweet potato flour and HMT-sweet potato flour and the deconvolution spectra in the range 1200 cm^{-1} to 800 cm^{-1}, respectively. Figure 1 shows that the peak distribution of sweet potato flour did not change after HMT, indicating that the structure type of the flour did not change. As seen from Figure 1 B, the absorbance ratio of the indicated HMT-flour peaks, 1047 cm^{-1}/1022 cm^{-1}, was lower than that of the original flour, and the ratio shows an overall downward trend with increasing water content, indicating that the short-range order of the flour was decreased. This may be because the synergistic actions of heat and water caused the part of starch that is easily gelatinize to swell in water because hydrogen bonds within and between molecular chains were broken, the double helix structure of amylopectin was unwound, the parallel structure was broken, and the degree of disorder increased (Wang et al., 2014; Chang et al., 2012). The absorbance ratio for 1022 cm^{-1}/998 cm^{-1} was higher than that of the original flour and shows an increasing trend with increasing water content, which indicates that the proportion of double helix structures inside the starch granules was increased after HMT. This may be because ordered molecules adopt new orientations and undergo rearrangement. The disordered ends of amylose in the starch particles were more tightly wound, which strengthened the interactions of molecular chain between the crystalline region and the amorphous region and strengthened the internal structure of the starch particles (Wang et al., 2017; Lawal, 2005). This was consistent with the abovementioned flour thermodynamic properties, in which the temperature and enthalpy change for starch pasting showed increases after HMT.

3.4 Crystallization characteristics

Starch particles are composed of crystalline regions and amorphous regions, and their crystallization characteristics can be characterized by X-ray diffraction patterns. The crystalline regions of starch granules show obvious peak diffraction characteristics in the X-ray diffraction pattern, which indicate that the region exhibits long range order, with relatively complete crystal types and large grain sizes. Amorphous regions show obvious dispersion diffraction characteristics in the X-ray diffraction pattern, indicating that this region exhibits long-range disorder and is also known

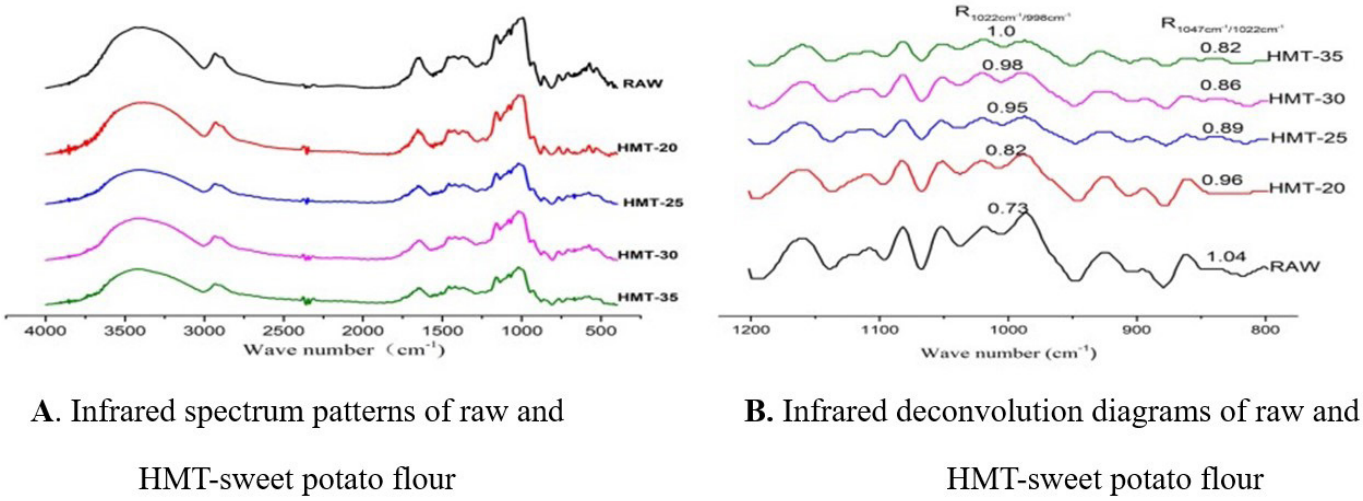

Figure 1. Infrared spectra of raw and HMT-sweet potato flour.

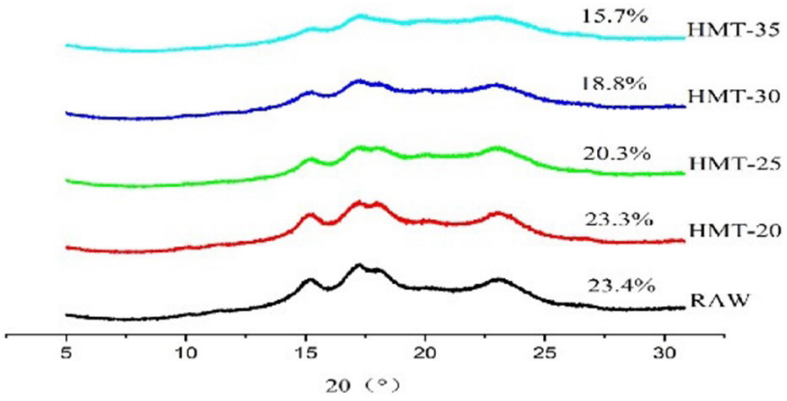

Figure 2. X-ray diffraction patterns of raw and HMT-sweet potato flour.

as an amorphous region (Wang et al., 2014; Zhang et al., 2018). As shown in Figure 2, the sweet potato flour before and after the HMT had strong diffraction peaks at 2θ = 15° and 23°, and there were undecomposed diffraction double peaks at 2θ = 17° and 18° that indicated that the sweet potato flour tested was a typical A-type starch and that the crystal type of the sweet potato flour did not change after the HMT. According to the changes in the crystallinity of the sweet potato flour before and after the HMT, which are listed on Figure 2, the crystallinity of the sweet potato flour after HMT had changed significantly; the extent of crystallinity was not only lower than that of the original flour but the crystallinity also showed a decreasing trend with increasing water content. This phenomenon is similar to the change in starch crystallinity in rice treated with HMT, as reported by Wang et al. (2018) and the change in starch crystallinity in rice treated with water and liquid, as reported by Barrera et al. (2013). This may be because starch granules swelled due to high temperature and water transport during HMT, hydrogen bonds broke within and between amyloid chains, and amyloid chains and double helices exhibited increased movement; this led to weaker interactions between the double helix structures at the ends of amylopectin and between straight and branched chains or the displacement and nonparallel rearrangement of double helix structures (Oliveira et al., 2018). It was also possible that at a high moisture level, some starch particles with loose structures swelled in water, the double helix structure at the end of amylopectin was untwisted and the starch underwent pasting and the loss of crystalline structure (Chen et al., 2015).

3.5 In vitro digestibility of sweet potato flour

Amylase causes a nonuniform reaction in starch systems, and the diffusion, absorption and digestion processes of the enzyme in starch can be simulated in vitro as is done for human gastrointestinal digestion (Wang et al., 2016). As shown in Table 3, the RDS, SDS and RS of sweet potato flour exhibited significant changes after HMT. The contents of RDS in HMT-20-35 were lower than that of the original flour, the contents of SDS and RS in HMT-20-35 were higher than that in raw powder. The variation rule of the digestive properties of sweet potato flour in Table 3 is consistent with the pasting properties and thermodynamic properties of sweet potato flour mentioned above. HMT-sweet potato flour had a more rigid structure and higher enthalpy and pasting temperature than untreated flour, which was consistent with the increase in SDS+RS content and the decrease in RDS. The high thermal energy in the humid and hot environment destroyed the original ordered structure of

Table 3. RDS, SDS and RS contents of raw and HMT-sweet potato flour.

Samples	*RDS(%)*	*SDS(%)*	*RS(%)*
RAW	52.73 ± 0.13^{a}	20.59 ± 0.43^{e}	26.68 ± 0.40^{d}
HMT-20	45.51 ± 0.13^{b}	24.20 ± 0.10^{c}	30.29 ± 0.18^{b}
HMT-25	30.62 ± 0.37^{e}	37.91 ± 0.18^{a}	31.47 ± 0.51^{b}
HMT-30	42.30 ± 0.25^{c}	30.27 ± 0.20^{b}	27.43 ± 0.15^{c}
HMT-35	43.97 ± 0.20^{d}	22.72 ± 0.23^{d}	33.31 ± 0.22^{a}

Values are means of three determinations (n = 3); values followed by different uppercase letters within a column differ significantly ($P < 0.05$).

flour particles to a certain extent, and the fracture of hydrogen bonds, migration of molecular chains, and unwinding and decomposition of the double helix decreased the crystallinity of starch (Ziegler et al., 2017). However, when the original ordered structure was destroyed by the pressure of water vapor and the rearrangement and orientation of the molecular chains, the molecular chains at the end of branched chains in the starch granules were intertwined more closely, the interaction forces between adjacent molecular chains and straight-branched chains were strengthened, and the rigidity of the starch was further strengthened (Martens et al., 2018). The dense accumulation of amorphous starch molecules and the compact rigid structure hindered the entry rate and decomposition rate of enzymes, which reduced digestibility (Ashwar et al., 2016). The increase in water content caused amylose to form an enzyme-resistant double helix structure with stable hydrogen bonds (Liao et al., 2019), which was consistent with the ratio of light absorption values for the aforementioned infrared bands at 1022 cm^{-1} and 998 cm^{-1} and consistent with the law determined by Sharma et al. (2015) and Barua & Srivastav (2017) for millet starch and mung bean starch. In the flour, the protein flakes attached to the surfaces of starch granules had a certain inhibiting effect on the diffusion of enzymes in the starch system, and the dense complex of amylose and lipids also hindered the infiltration of enzymes into the starch to some extent (Ye et al., 2018).

4 Conclusions

The results obtained in this study suggest that the changes in physicochemical properties and digestive properties of sweet potato flour were mainly caused by the reconfiguration of the internal structure of flour particles caused by heat-moisture treatment. The HMT-sweet potato flour not only has the characteristics of low digestibility and low viscosity similar to HMT-sweet potato starch, but also has good cold and heat stability.

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