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Differences in physicochemical properties and *in vitro* digestibility between tartary buckwheat flour and starch modified by heat-moisture treatment

Yao Xiao [a], Hang Liu [b], Ting Wei [a], Jing Shen [a], Min Wang [a,] *

[a] College of Food Science and Engineering, Northwest A & F University, Yangling, Shaanxi 712100, PR China
[b] School of Life Sciences and Biotechnology, Shanghai Jiao Tong University, Shanghai Center for Systems Biomedicine, Shanghai 200240, PR China

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A B S T R A C T

Native tartary buckwheat starch (NTBS) and native tartary buckwheat flour (NTBF) were modified by heat-moisture treatment (HMT) with different moisture levels (200, 250, 300, and 350 g/kg) to investigate the effect of HMT on their physicochemical properties and *in vitro* digestibility. Compared to native samples. The onset, peak, conclusion temperatures of HMT-modified samples increased, while the pasting viscosities, enthalpy decreased. After HMT, the gel hardness of starch and flour decreased by 262 and 62.1 g, respectively. But the slowly digestible starch (SDS) content of starch and flour increased by 71.3 and 27 g/kg, respectively, meanwhile the resistant starch (RS) content of starch and flour increased by 23.8 and 41 g/kg, respectively. HMT did not change the A type crystalline pattern of NTBS and NTBF, but the relative crystallinity of NTBS and NTBF increased by 16% and 7%, respectively. HMT had a far greater effect on morphology and pasting properties on flour than on starch. The granule morphology was noticeably changed by HMT, HMT created more cavities and holes on the surface of HMT flours than HMT starches. The results showed that HMT greatly modified the physicochemical properties and *in vitro* digestibility of NTBS and NTBF to expand their application range.

1. Introduction

Tartary buckwheat (*Fagopyrum tataricum*) belongs to the genus *Fagopyrum*, Polygonaceae family, which is one of the main buckwheat species cultivated around the world (F. Zhu, 2016). Tartary buckwheat has high resistance to the stress of cold, dry and harsh climate, so that it can grow in high altitudes with low precipitation and low temperature (Liu, Lv, Peng, Shan, & Wang, 2015b; F. Zhu, 2016). Although tartary buckwheat has strong bitter taste, it has been popularly consumed in European and Asian countries for a long time. As a potential functional food source, tartary buckwheat has been concerned based on its bioactive nutrients and multiple health benefits (Ahmed et al., 2013; Guo et al., 2011). Many functional foods derived from tartary buckwheat have been commercialized, including buckwheat enhanced breads, noodles, tea, biscuits, sprouts and finally buckwheat honey (Giménez-Bastida, Piskula, & Zieliñski, 2015).

Heat-moisture treatment (HMT) is an important physical modification technique applied to starch for partly altering its physicochemical characteristics and properties to meet the multiple processing requirements in food production. HMT is a low-cost method without any residual chemical reagents, and it is safe and suitable for food applications. Numerous studies have demonstrated that there is a major influence of HMT on the structure, physicochemical properties, as well as *in vitro* digestibility of starches isolated from different botanical sources, including significant changes in crystallinity, chain interaction, granule swelling, pasting characteristics, granules morphology, enzyme hydrolysis, textural and thermal properties (BeMiller & Huber, 2015). Compared to native starch, HMT-modified starches exhibit an enhancement in gelatinization temperature, a broadened gelatinization temperature range, an increased mobility to starch chains and helical structures but decreased enthalpy of gelatinization

Abbreviations: NTBS, native tartary buckwheat starch; NTBF, native tartary buckwheat flour; HMT, heat-moisture treatment; AAC, apparent amylose content; RC, relative crystallinity; db, dry weight basis; RDS, rapidly digestible starch; SDS, slowly digestible starch; RS, resistant starch.

* Corresponding author.
E-mail addresses: changyangxiaoyao@163.com (Y. Xiao), lhhyyf123@163.com (H. Liu), weiting1123@163.com (T. Wei), s_shenjing@126.com (J. Shen), wangmin20050606@163.com (M. Wang).

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(Gunaratne & Hoover, 2002; R. Hoover, 2010; Shin, Kim, Ha, Lee, & Moon, 2005; H. Singh, Chang, Lin, Singh, & Singh, 2011; Vermeylen, Goderis, & Delcour, 2006). Liu et al. (2015a) and Liu et al. (2015b) reported that HMT-modified common and tartary buckwheat starches had higher gelatinization temperature and relative crystallinity, but lower peak viscosity, enthalpy, solubility and swelling power. Besides the physicochemical properties, HMT also can affect the digestibility of starch. Several studies have investigated the effect of HMT on the digestibility of rice starch and wheat starch (Chen, He, Fu, & Huang, 2015; Puncha-arnon & Uttapap, 2013), these results showed that HMT has a marked impact on the starch digestibility.

Flours, which are obtained by grinding and sifting the starch-containing plant organelles, are powdery materials for general foods including bread, biscuits and noodles (Puncha-arnon & Uttapap, 2013). Tartarty buckwheat flour (TBF) contains beneficial and functional components, including poly-un saturated essential fatty acid, D-chiro-inositol, fagopyritols and flavonoids (F. Zhu, 2016), making TBF become a good basis and have beneficial effect for diet. However, because of lacking gluten, the applications of native TBF have been limited.

A few studies have identified that the HMT significantly altered the properties of flours. Both Ahn et al. (2013) and Chen et al. (2015) successfully utilized HMT to increase slow digestible starch and resistant starch fraction of sweet potato and wheat flour, respectively. However, some of the results were inconsistent. The relative crystallinity and enthalpy of HMT-modified wheat flour increased, but the relative crystallinity and enthalpy of HMT-modified sweet potato flour decreased. After HMT treatment, the X-ray diffraction pattern of sweet potato flour maintained the A-type, but it transferred from A to A + V type when wheat flour modified by HMT(Ahn et al., 2013; Chen et al., 2015). In addition, HMT-modified rice and sorghum flours/starches have been investigated, and the results showed that HMT had a greater effect on thermal and viscosity properties of rice and sorghum flours than those of rice and sorghum starches (Puncha-arnon & Uttapap, 2013; Sun, Han, Wang, & Xiong, 2014).

Previous studies on buckwheat starch showed that high hydrostatic pressure and annealing could significantly alter the physicochemical properties and *in vitro* digestibility of both common and tartary buckwheat starch (Liu, Wang, Cao, Fan, & Wang, 2016b; Liu et al., 2015a, 2016a, 2016c). The HMT could modify the physicochemical properties of common and tartary buckwheat starch, and improve the *in vitro* digestibility of common buckwheat starch (Liu et al., 2015a, 2015b), but it is seldom reported HMT altered the *in vitro* digestibility of tartary buckwheat starch (TBS), as far as we know. In addition, the effect of HMT on the physicochemical properties and *in vitro* digestibility of TBF has not been investigated, and the differences in physicochemical properties and *in vitro* digestibility between TBF and TBS treated by HMT have not yet reported.

This study aims to obtain the information on pasting, thermal, textural properties and *in vitro* digestibility of HMT-modified TBF, as well as to investigate the differences in physicochemical properties and *in vitro* digestibility between TBF and TBS modified by HMT. Meanwhile, the relevant results can be used to extend the application of tartary buckwheat for positive health benefit.

2. Materials and methods

2.1. Materials

Tatary buckwheat seeds (Xinong 9940) were obtained from Hongsheng company (Yulin, Shaanxi, China) in 2015. Tartary buckwheat grains were obtained by milling the seeds with a dehuller (K300, LiangYou machinery manufacture Co., Ltd, Shijiazhuang, China). The α-amylase from porcine pancreas (A3176, 250.05 nKat/mg, Solid), pepsin from porcine gastric mucosa (P7215-100G, purity≥1%, ≥6668 nKat/mg) and amyloglucosidase (A9913, 55011 nKat/ml) were purchased from Sigma- Aldrich Chemical Co. (St. Louis, MO, USA). All the chemicals used in this study which are purchased from Xilong Chemical Co., Ltd (Shantou city, Guangdong province, China), were analytical grade.

2.2. Native tartary buckwheat flour preparation

NTBF was obtained by grinding the dehulled grains with a disintegrator (FW100, Tianjin TAISITE instrument Co., Ltd, Tianjin, China), and the flour was passed through a 0.15 mm sieve and stored at 4 °C for future research.

2.3. Native tartary buckwheat starch isolation

The starch was isolated from dehulled tartary buckwheat grains by using a modified procedure (Liu et al., 2015b). The dehulled grains were steeped in distilled water with 330 g/L for 2 h, then ground for 1 min with a soybean milk machine (JYZ-E6, JoYoung Co., Ltd, Jinan, Shandong, China), and passed through 0.15 mm sieve. The filtrate kept for 24 h and removed the supernatant. The sediment was dried at 45 °C in a constant temperature convection oven (DHG-9203A, Shanghai Jinghong Laboratory instrument Co., Ltd, Shanghai, China). Subsequently, the dried precipitate was suspended into 3 g/L sodium hydroxide solution with a final concentration of 100 g/L and kept for 24 h. After decanting the cloudy supernatant, the starch layer was washed with distilled water and the washing step was repeated until without yellow color. The starch sediment was re-suspended into distilled water and passed through a 0.075 mm sieve. The filtrate was allowed to stand at room temperature for 12 h and the supernatant was drained, then the starch cake was dried at 45 °C for 12 h. The isolated NTBS was stored at 4 °C for future investigation.

2.4. Chemical composition of starch and flour

AOAC methods (1990) were used for the determination of moisture, ash, and lipid contents. Protein content was determined by a Kjeltec Auto Analyzer (VS-KT-P, MRK Co., Japan) with a conversion factor of 5.89. Total starch was determined by the method of S. Singh, Raina, Bawa, and Saxena (2005). Apparent amylose content (AAC) was determined according to the method of Juliano et al. (1981), and the AAC of NTBS and NTBF was calculated with respect to dry starch and dry flour, respectively.

2.5. Heat-moisture treatment

The HMT was performed based on the method of Liu et al. (2015a) with some modifications. The specific moisture level of TBF and TBS was adjusted to 200 g/kg, 250 g/kg, 300 g/kg, and 350 g/kg by adding distilled water respectively and sealed in containers. Then all the samples were equilibrated for 24 h at room temperature before heating at 110 °C for 16 h. After cooling to room temperature, all the samples were air-dried at 45 °C overnight in the convection oven. According to moisture level, HMT-modified TBS samples were referred to as NTBS, TBS-20, TBS-25, TBS-30 and TBS-35, and the HMT-modified TBF samples were referred to as NTBF, TBF-20, TBF-25, TBF-30 and TBF-35.

2.6. Scanning electron microscopy

The morphology of starch and flour samples was observed by scanning electron microscopy (SEM; JEOL JSM-6360LV, JEOL Ltd.,