**In vitro** digestibility and changes in physicochemical and structural properties of common buckwheat starch affected by high hydrostatic pressure

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**Abstract**

High hydrostatic pressure (HHP), a non-thermal processing technology, was applied at 120, 240, 360, 480, and 600 MPa to assess its effect on the **in vitro** digestibility, physicochemical, and structural properties of common buckwheat starch (CBS). HHP treatment resulted in CBS granules with more rough surfaces. With the increasing pressure level, amylose content, pasting temperature, and thermal stability substantially increased and relative crystallinity, hardness, swelling power, and viscosity decreased. At 120–480 MPa, HHP did not affect the 'A'-type crystalline pattern of CBS. However, at 600 MPa, HHP contributed to a similar 'B'-type pattern. Compared with native starch, HHP-modified CBS samples had lower **in vitro** hydrolysis, reduced content of rapidly digestible starch, and increased levels of slowly digestible starch and resistant starch. These results revealed that the **in vitro** digestibility, physicochemical, and structural properties of CBS are effectively modified by HHP.

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**1. Introduction**

According to epidemiological studies, the regular consumption of minor cereal or pseudocereal products is positively correlated with the reduced risk of several chronic diseases, including type II diabetes, cardiovascular disease, obesity, and cancer (Jones et al., 2000; Kaur, Jha, Sabikhi, & Singh, 2014). Common buckwheat (*Fagopyrum esculentum* Moench), which belongs to the genus *Fagopyrum* of the family Polygonaceae, is one of the two main species of buckwheat that has been consumed in China for 2000 years (Li & Zhang, 2001). Due to its high nutritional content (e.g., antioxidants, dietary fiber, protein and resistant starch) and disease-preventative roles, common buckwheat has recently become a major research focus (Ahmed, Khalid, Ahmad, Abbasi, Latif, & Randhawa, 2014; Liu, Guo, et al., 2015; Liu, Lv, Peng, Shan, & Wang, 2015; Qin, Wang, Shan, Hou, & Ren, 2010). However, common buckwheat is devoid of gluten, which limits its application as food thickener, bulking agent, gelling agent, colloid stabilizer, and so forth (Li & Zhang, 2001). It has been reported that the modification of starch can improve its process properties, meeting the requirements of food processing (Zavarezee & Dias, 2011). Therefore, in order to overcome the shortcomings of common buckwheat, common buckwheat starch (CBS) may be modified to alter its physicochemical properties to obtain wider applications.

As a non-thermal processing technology, high hydrostatic pressure (HHP) treatment, which is performed at room temperature and 100–1000 MPa to sterilize and modify the packed materials in a vessel (Tian, Li, Zhao, Xu, & Jin, 2014), is suitable for production of minimally processed foods. The potential applications of HHP in food processing and preservation have been investigated as an alternative to traditional hydrothermal treatments (Vallons & Arndt, 2009). In recent years, the effects of HHP on the physicochemical properties of more than 25 starches, including those from wheat, corn, rice, barley, potato, canna, pea, lentil, mung bean, peanut, rye, taro, amaranth, kozou, arrow root, tapioca, sorghum, and faba bean, have been evaluated (Kim, Kim, & Baik, 2012). The studies have reported that HHP treatment decreases swelling index, weaken gels (Stute, Klingler, Boguslawski, Eshtakihi, & Knorr, 1996), lowers the gelatinization temperature and gelatinization enthalpy (Kawai, Fukami, & Yamamoto, 2012), decreases...
the susceptibility to amylolytic enzymes (Douzals, Perrier Cornet, Gervais, & Coquille, 1998), and affects the crystalline structure and morphology of starch (Blaszczyk, Valverde, & Fornal, 2005). Ahmed, Ramaswamy, Ayad, Ali, and Alvarez (2007) reported that ‘A’-type starches are most sensitive to HHP treatment, followed by ‘C’-type and ‘B’-type starches. In addition to starch type, there are other factors that affect HHP treatment including pressure level, treatment time, and water content (Liu, Selomulyo, & Zhou, 2008). However, there are few reports on how HHP affects the physicochemical properties of CBS.

Digestibility is another important property of starch that can be affected by HHP treatment. Based on its hydrolysis rate, starch is classified into three fractions: rapidly digestible starch (RDS, digested within 20 min), slowly digestible starch (SDS, digested between 20 and 120 min), and resistant starch (RS, indigestible) (Englyst, Kingman, & Cummings, 1992). RDS induces a sudden rise in blood glucose levels, whereas SDS results in a gradual increase in blood glucose levels, which is important in the prevention of diabetes and cardiovascular diseases (Lehmann & Robin, 2007). RS decreases serum cholesterol levels, suppresses gall stone formation, and prevents certain types of cancer (Asp, Van Amelsvoort, & Hautvast, 1996). Therefore, SDS and RS have significant health benefits. Following HHP treatment, SDS and RS contents increase in wheat, corn, potato, quinoa, and normal and waxy rice starches (Linsberger-Martin, Lukaš, & Berghofer, 2012; Stute, Klingler, Boguslawski, Eshtighi, & Knorr, 1996), but there is little evidence of the HHP on CBS.

Therefore, this study was performed to assess the effects of different HHP pressure levels on the in vitro digestibility, physicochemical, structural, and textural properties of CBS. The relevant results will be relevant for further research and CBS applications.

2. Materials and methods

2.1. Materials

Common buckwheat seeds (Yuqiao #4) were harvested in Yulin (Shaanxi Province, China) in 2014. The moisture content of the native seeds was 13.8%. Standard amylase (A0521; purity ≥70%, from potato) and amylpectin (A8515; from potato), porcine pancreatic α-amylase (A3176; 22 U/mg), amyloglucosidase (A9913; 3300 U/mL), and pepsin from porcine-stomach mucosa (P7125; purity ≥1%, ≥400 U/mg) were purchased from Sigma Chemical Company (St. Louis, MO, USA). All chemicals and reagents were of analytical grade.

2.2. Starch isolation

Common buckwheat starch was isolated according to the method described by Liu, Guo, et al. (2015) and Liu, Lv, et al. (2015).

2.3. HHP treatment

HHP treatment was performed according to the method reported by Blaszczyk, Fornal, Valverde, and Garrido (2005) and Blaszczyk, Valverde, et al. (2005), with minor modifications. The moisture level of CBS was adjusted to 20% (w/v) by adding appropriate volumes of distilled water. The sample was vacuum-packed in 100-ml polyethylene bags with a vacuum sealer (XZ-500D, Shanghai Xiangzheng Machine Co., Ltd., Shanghai, China), allowed to equilibrate for 24 h at 4 °C, and subjected to different pressures levels (120, 240, 360, 480, and 600 MPa) for 20 min at room temperature using a high-pressure instrument (HPP L2-600/2, Hua Tai Sen Miao Ultra-pressure Equipment Co., Ltd., Tianjin, China). The pressure-transmitting medium was distilled water, and the maximum working pressure was 600 MPa. Following HHP treatment, the sample bags were opened, vacuum-filtered, and freeze-dried. The HHP-modified CBS samples were labeled according to the corresponding pressure level (HHP-120, HHP-240, HHP-360, HHP-480, and HHP-600).

2.4. Chemical composition of samples

Amylose content (AMC) was determined by an iodine-binding procedure (Liu, Lv, et al., 2015). Protein content was determined by Kjeldahl (N × 5.89) in a Kjeltec Auto Analyzer (Foss Tecator AB, Höganas, Sweden). Total starch was measured by the method reported by Singh, Raina, Bawa, and Saxena (2005). Moisture, ash, and crude lipid contents of samples were determined according to AOAC methods (2000).

2.5. Morphological properties

The morphology of native and HHP-modified CBS was examined by scanning electron microscopy (SEM; JSM-6300, JEOL Ltd., Tokyo, Japan) at an acceleration potential of 20 kV. The starch samples were mounted on a double-sided adhesive tape attached to a metal stub and coated with 20 nm gold under vacuum before being observed under the SEM.

2.6. Swelling power and solubility

The swelling power and solubility of native and HHP-modified CBS were determined by the method reported by Choi, Kim, Park, Kim, and Baik (2009). Starch (50 mg) was transferred into dry centrifugal tubes, weighed, and mixed with distilled water (5 mL). The tubes were incubated in a shaking water bath at 50, 60, 70, 80, and 90 °C for 30 min, cooled to room temperature, and centrifuged at 657 × g for 15 min. The supernatant was carefully decanted, and the resulting tubes with their contents were weighed. The residue obtained after drying the supernatant represented the amount of starch dissolved in water. Solubility and swelling power were calculated on a dry weight basis (db) using the following equations:

\[
\text{Solubility} = \frac{\text{the weight of dried supernatant}}{\text{weight of starch}}
\]

\[
\text{Swelling power} = \frac{W_2 - W_1}{\text{weight of starch}}
\]

where \(W_1\) represents the weight of the tube with starch sample, and \(W_2\) represents the weight of the tube with the resulting precipitate after decanting the supernatant.

2.7. X-ray diffraction (XRD)

The XRD patterns of native and HHP-modified CBS were obtained from a D/Max-2200 X-ray diffractometer (Rigaku Denki Co., Tokyo, Japan). The sample was pretreated overnight at room temperature in a chamber with saturated relative humidity and scanned at 5°–60° (2θ) at a rate of 4°/min, a target voltage of 40 kV, and a current of 30 mA. The XRD patterns were compared to the peak characteristics of a theoretical diffractogram (Zobel, Young, & Rocca, 1988). Relative crystallinity (RC) was calculated as the ratio between the crystalline areas and the amorphous regions of the X-ray diffractograms (Nara & Komiya, 1983).

2.8. Textural properties

A TA-XTPLUS/50 Textural Analyzer ( Stable Micro Systems, Godalming, UK) was used to determine the textural properties of native and HHP-modified CBS gels. A starch suspension (10%) was transferred into a container and heated in a boiling water
bath for 30 min. After cooling, it was stored overnight at 4°C. The resulting gel was penetrated to a distance of 10 mm at 1 mm/s using a flat cylindrical probe (5 mm diameter). The penetration was repeated twice to generate a force–time curve. Hardness, cohesiveness, springiness, gummy ness, and chewiness were computed.

2.9. Differential scanning calorimetry (DSC)

A differential scanning calorimeter (STA409PC; Netzsch Co., Ltd., Selb, Germany) was used to determine starch gelatinization characteristics. Starch (3 mg, db) was weighed into an aluminum pan and mixed with distilled water at a ratio of 1:3.5 (w/w). To ensure sample equilibration, the pan was sealed and stored at room temperature for 24 h. Subsequently, the sample was heated from 30 to 150°C at 10°C/min. Onset temperature ($T_o$), peak temperature ($T_p$), conclusion temperature ($T_c$), and gelatinization enthalpy ($\Delta H$) were calculated.

2.10. Pasting properties

The pasting properties of native and HHP-modified CBS were evaluated using a Rapid Visco Analyzer (RVA-4, Newport Scientific Co., Ltd., Warrewood, New South Wales, Australia). Starch (3 g, db) was dispensed in distilled water (25 g) in an aluminum RVA sample canister. The programmed cycle was set at 50°C for 1 min, increased to 95°C in 3.7 min, held at 95°C for 2.5 min, decreased to 50°C in 3.8 min, and held at 50°C for 2 min. Pasting temperature (PT), peak viscosity (VF), breakdown viscosity (BD), setback viscosity (SB), and final viscosity (VF) were calculated.

2.11. Starch digestibility

2.11.1. In vitro digestion of starch

The in vitro digestibility of native and HHP-modified CBS was determined with the method reported by Liu, Guo, et al. (2015) and Liu, Lv, et al. (2015).

2.11.2. Resistant starch content

The RS content was calculated by the method described by Liu, Guo, et al. (2015) and Liu, Lv, et al. (2015) without any modifications.

2.12. Statistical analyses

The data were statistically analyzed by one-way analysis of variance (ANOVA) using SPSS version 19.0 software for Microsoft Windows (SPSS Inc., Chicago, IL, USA). Triplet determinations were performed to obtain mean values and standard deviations. The least significant difference (LSD) test was performed to determine differences among treatments. Statistical significance was set at $p < 0.05$.

3. Results and discussion

3.1. Chemical composition of CBS

The chemical composition of native and HHP-modified CBS is presented in Table 1. Moisture content ranged from 10.5% to 11.2%. Following HHP treatment, moisture content of the samples decreased, especially in HHP-600. The reduction in moisture content after HHP treatment might be attributed to changes in starch granule structure, which facilitated the loss of free water. The protein content of CBS samples slightly decreased from 0.35% (native starch) to 0.32% (HHP-600), and there was no significant difference among the values. This reduction in protein content was due either to the association between protein and starch molecules (e.g., via hydrogen, covalent, and ionic bonds) or the formation of a protein-starch complex (Adebowale, Afolabi, & Olu-Owolabi, 2005). AMC significantly increased following HHP treatment, among which increased by 2.3% (HHP-120), 3.2% (HHP-240), 4.3% (HHP-360), 5.1% (HHP-480), and 7.3% (HHP-600). This increase in AMC, which was positively correlated with pressure level, was due to limited amylose leaching as a result of interactions between amylose-amylopectin and amylose-lipid during HHP treatment (Oh, Hemar, Anema, Wong, & Pinder, 2008). The HHP-induced degradation of amylopectin might be another reason for the increase in AMC. HHP resulted in interactions between lipid and amylose, which limited the mobility of crude lipid and reduced crude lipid content. A reduction in the inner diameter of the amylose helical structure and the formation of lipid-starch complexes during HHP treatment were the main factors that contributed to the decrease in total starch content.

3.2. Morphological properties

Native CBS granules had irregular oval, spherical, or polygonal shapes with smooth surfaces and no cavities or fissures (Fig. 1A). On the other hand, cavities were present on the surface of HHP-120 and HHP-240 (Fig. 1B and C), and fissures and deep holes were observed on the surfaces of HHP-360 (Fig. 1D). The shapes of CBS granules remained intact after HHP treatment at 120–360 MPa; however, HHP-480 granules collapsed and acquired a doughnut shape (Fig. 1E), which is the typical granular structure upon pressure gelatinization (Douzals, Perrier Cornet, Gervais, & Coquelle, 1998). HHP-600 granules gelatinized, swelled, deformed, collapsed, and then coalesced (Fig. 1F).

Our findings revealed that the effect of HHP on CBS morphological properties was pressure dependent. HHP resulted in strong interactions between amylose and amylopectin chains, which contributed to a compact starchy structure with the presence of cavities, fissures, and holes on the surface (Blaszczyk, Fornal, et al., 2005). Blaszczyk, Fornal, et al. (2005) and Blaszczyk, Valverde, et al. (2005) reported that the outer section of starch granules mainly consists of amylopectin with a wide range of high-molecular mass fragments, while the interior section mainly consists of a gel-like
network, which explains why most of the morphological changes induced by HHP occurred in the interior of the CBS granules. These observations were in good agreement with previous studies on rice, maize, waxy corn, and potato starches (Katopo, Song, & Jane, 2002; Li, Bai, Mousaa, Zhang, & Shen, 2012; Liu, Selomulyo, & Zhou, 2008).

3.3. Swelling power and starch solubility

The effect of temperature on swelling power and solubility of native and HHP-modified CBS is presented in Fig. 2. As temperature increased from 50 to 90 °C, swelling power and solubility of all starch samples increased (Fig. 2A and B). At 50–60 °C, HHP-600 had higher swelling power and solubility than those of other samples; however, results were opposite at higher temperatures. Compared to native CBS, HHP-modified CBS had reduced swelling power and solubility at 70–90 °C. This reduction was positively correlated with pressure levels. Similar results have been reported with rice and corn starches (Kim, Choi, Kim, & Baik, 2010; Li, Bai, Mousaa, Zhang, & Shen, 2012).

Swelling power and solubility represent the degree of interaction among starch chains within the amorphous and crystalline regions of the granules (Singh & Kaur, 2004). This interaction is influenced by several factors, e.g., amylose-to-amylopectin ratio, molecular weight or distribution, length of branching, and conformation (Hoover, 2001). New crystals created by amylose and lipids at 50–60 °C inhibited excessive swelling of granules (Mandala & Bayas, 2004). The higher swelling power of HHP-modified CBS samples at lower temperatures demonstrated the presumed aggregation of amylose molecular under pressure. These amylose aggregates prevented lipid–starch linkages at 50–60 °C and induced greater water retention.

The reduction in swelling power and solubility of HHP-modified CBS samples at 70–90 °C might be due to the rearrangement of starch molecules, which limited the hydration and swelling capacity of CBS. Partially or completely disintegrated starch granules inhibited the solubilization of amylose, thereby limiting swelling power and solubility of CBS (Stolt, Oinonen, & Autio, 2001). Furthermore, the formation of amylose-lipid complexes in CBS granules during HHP modification might have limited the mobility of soluble amylose molecules, thereby inhibiting swelling power and solubility of CBS (Katopo, Song, & Jane, 2002; Oh, Hemar, Anema, Wong, & Pinder, 2008). Even though reductions in starch swelling power and solubility following HHP treatment have been studied, further research is required to understand how HHP affects solubility and swelling of starch.
3.4. XRD pattern and RC

The XRD patterns and RC of native and HHP-modified CBS are shown in Fig. 3. Based on the diffraction peaks at 2θ values of 15.22°, 17.32°, 18.14°, and 23.12°, native CBS exhibited a typical ‘A’ type crystalline pattern. Compared with native starch, HHP-modified CBS samples (120, 240, 360, and 480 MPa) had diffraction peaks at similar diffraction angles with decreased intensity. However, HHP-600 underwent a distinct transformation from ‘A’-type toward a ‘B’-type-like pattern, showing a strong peak at 17° and few weak peaks at around 20°, 22°, and 24° (Liu et al., 2009). Similar findings have been reported in pressurized rice, maize, and wheat starches (Katopo, Song, & Jane, 2002; Stute, Klingler, Boguslawski, Eshtiaghi, & Knorr, 1996).

The RC of HHP-modified CBS ranged from 26.2% to 37.8% in the order, HHP-120 > HHP-240 > HHP-360 > HHP-480 > HHP-600. These values, especially those of HHP-480 and HHP-600, were lower than that of native starch (Fig. 3). These results revealed that RC decreased with increasing pressure level. Additionally, some granules began to gelatinize under low pressure (120 MPa). The ‘A’-type starch, had a scattered amylpectin structure, which was flexible and easily destroyed, allowing a rearrangement of double helices that resulted in changes in the crystalline structure. This structure was attributed to the reduction in RC under pressurization (Jane, Wong, & McPherson, 1997). In addition, water molecules entered the starch granules during HHP treatment, causing the starch double helices in the crystalline region to open and to become more fragile (Kawai, Fukami, & Yamamoto, 2012), thereby decreasing the RC. High pressure allowed CBS granules to react with water molecules, resulting in a change in the XRD pattern. HHP at 600 MPa completely disrupted the crystalline structure of CBS.

3.5. Textural properties

The textural properties of native and HHP-modified CBS samples are shown in Table 2. HHP treatment progressively reduced hardness, adhesiveness, gumminess, and chewiness of starch gels. There was no remarkable difference in springiness among the samples. Cohesiveness ranged from 0.415 (HHP-600) to 0.652 (native starch). Hardness declined significantly from 148.7 (native starch) to 22.3 (HMT-600).

Starch gel formation mainly depends on the amount of swollen starch granules. A reduction in the levels of leached amylose plays an important role in reducing gel hardness (Puncha-arnon & Uttapap, 2013). Therefore, during gelatinization, the HHP-modified CBS with higher AMC swelled less than native starch, thereby resulting in weaker gels. These findings were consistent with the results presented in Table 1 and Fig. 3. Coupled to the amylpectin structure, poor water–starch and starch–starch molecular interactions were other contributors to the weaker textural properties of HHP-modified starch gels (Vittadini, Carini, Chiavaro, Rovere, & Barbanti, 2008). The results revealed that the characteristics of CBS gels can be significantly altered by HHP.

3.6. DSC

Compared with native starch, HHP-modified CBS had progressively lower values of Tg, Tp, Tc, Tc − Tg (ΔT), and ΔH. The value of Tg significantly decreased from 65.5° C (native starch) to 61.6° C (HHP-600), and ΔH decreased from 22.5 J/g (native starch) to 8.6 J/g (HHP-600). These reductions were positively correlated with pressure level (Table 3), indicating different degrees of gelatinization following HHP treatment. Similar results have been reported with wheat, rice, corn, and potato starches (Kim, Choi, Kim, & Baik, 2010; Li, Bai, Mousaa, Zhang, & Shen, 2012; Muhr & Blanshard, 1982).

HHP treatment destabilized the crystalline lamella and decreased the crystallinity. As a result, less energy was required for gelatinization, contributing to a reduction in gelatinization temperature (Blaszcak, Valverde, et al., 2005). According to Kaur, Singh, Ezekiel, and Sodhi (2009), ΔT represents the stability of the
crystalline region in the starch granule and is positively correlated with crystallinity. Gunaratne and Hoover (2002) demonstrated that the $\Delta T$ value of starches is related to the strength of heterogeneous crystals. The narrow $\Delta T$ values in Table 3 indicated that HHP treatment significantly disrupted the crystalline structure. As a result, crystalline regions of HHP-modified granules had lower stability. These results were in good agreement with results from XRD analysis. The $\Delta H$ value calculated by DSC is mainly related to the disruption of double helices (Miao, Zhang, & Jiang, 2009). The reduction in $\Delta H$ of HHP-modified samples was attributed to the gelatinization of some amyllose molecules. The destruction of double helices and the ordered structure of crystalline region of CBS granules might also explain the reduction of $\Delta H$. DSC results indicated that HHP treatment significantly modified the gelatinization properties of CBS.

### 3.7. Pasting properties

Viscosity affects the applications of starch in food industry. The pasting parameters of native and HHP-modified CBS samples are summarized in Table 4. HHP-modified samples displayed higher pasting temperatures compared to that of native starch, with HHP-600 reaching the highest value (68.8 °C). This increase in PT of HHP samples was consistent with the pressure level. Compared with native starch, HHP-modified CBS samples had significantly lower viscosity, including PV, BD, SB, and FV. The PV significantly decreased from 4019 (native starch) to 371 (HHP-600), and SB decreased from 1915 (native starch) to 347 (HHP-600). These reductions were negatively correlated with pressure level. The results indicated that HHP treatment significantly improved the thermostability of CBS, in agreement with previous findings on waxy corn, tapioca, rice, potato, canna, and arrow root starches (Stute, Klingler, Boguslawski, Eshtiaghi, & Knorr, 1996). Structure changes induced by HHP (e.g., transformation of crystalline structure, formation of amylose-lipid complexes, loss of molecules order, and intertwined amyllopectin) limited amylose leaching and amylopectin dispersion of CBS, increased pasting temperature, and reduced viscosities (Hoover, Hughes, Chung, & Liu, 2010).

The reduction in viscosity following HHP treatment mainly results from restricted swelling of starch granules. Li et al. (2012) observed that PV is indicative of early and rapid swelling of starch granules along with amylose leaching, and FV represents the stability of gelatinized paste. The reduction in PV suggested that HHP limited hydration and swelling of CBS granules during gelatinization. The reduction in BD might also be attributed to the limited granular swelling. HHP-modified CBS was stable during continued heating and shearing, reducing the destabilization effect on the amorphous region of melting crystallites and leading to a higher pasting temperature (Gunaratne & Hoover, 2002). The changes in SB revealed the gelling ability and retrogradation tendency of CBS. A marked reduction in amylose leaching played a role in the decrease in SB, especially in the starch samples with high AMC (Lan et al., 2008). More stable conformation of amylose-lipid complexes in CBS granules with HHP treatment might be another main reason for the decrease in SB (Kim, Kim, & Baik, 2012). Therefore, HHP is an effective method for modifying the pasting properties of CBS.

### Table 2

Textural properties of native and HHP-modified starches.

<table>
<thead>
<tr>
<th>Cultivars</th>
<th>Hardness (g)</th>
<th>Cohesiveness (g)</th>
<th>Adhesiveness (g)</th>
<th>Springiness</th>
<th>Gumminess (g)</th>
<th>Chewiness (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Native</td>
<td>148.7 ± 0.12a</td>
<td>0.652 ± 0.09a</td>
<td>146.5 ± 0.35a</td>
<td>0.96 ± 0.01a</td>
<td>96.9 ± 0.02a</td>
<td>93.0 ± 0.08a</td>
</tr>
<tr>
<td>HHP-120</td>
<td>98.3 ± 0.21b</td>
<td>0.582 ± 0.21b</td>
<td>142.5 ± 0.28a</td>
<td>0.98 ± 0.01a</td>
<td>57.2 ± 0.04b</td>
<td>56.1 ± 0.11b</td>
</tr>
<tr>
<td>HHP-240</td>
<td>87.5 ± 0.16c</td>
<td>0.557 ± 0.04b</td>
<td>85.1 ± 0.15b</td>
<td>0.03 ± 0.01a</td>
<td>48.7 ± 0.09c</td>
<td>45.3 ± 0.04c</td>
</tr>
<tr>
<td>HHP-360</td>
<td>52.1 ± 0.23d</td>
<td>0.526 ± 0.22bc</td>
<td>81.9 ± 0.04b</td>
<td>0.94 ± 0.07a</td>
<td>27.4 ± 0.11d</td>
<td>25.8 ± 0.01d</td>
</tr>
<tr>
<td>HHP-480</td>
<td>40.1 ± 0.02e</td>
<td>0.502 ± 0.03c</td>
<td>68.7 ± 0.21c</td>
<td>0.96 ± 0.13a</td>
<td>20.1 ± 0.24e</td>
<td>19.3 ± 0.26e</td>
</tr>
<tr>
<td>HHP-600</td>
<td>22.3 ± 0.12f</td>
<td>0.415 ± 0.07d</td>
<td>27.6 ± 0.02d</td>
<td>0.98 ± 0.12a</td>
<td>9.3 ± 0.03f</td>
<td>9.1 ± 0.18f</td>
</tr>
</tbody>
</table>

Means of triplicate determination ± S.D. with the different letter in a column within each property are significantly different ($p < 0.05$).

### Table 3

Gelatinization characteristics of native and HHP-modified starches.

<table>
<thead>
<tr>
<th>Samples</th>
<th>$T_g$ (C)</th>
<th>$T_m$ (C)</th>
<th>$T_p$ (C)</th>
<th>$\Delta T$ (C)</th>
<th>$\Delta H$ (J/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Native</td>
<td>65.5 ± 0.05a</td>
<td>76.5 ± 0.11a</td>
<td>80.3 ± 0.11a</td>
<td>14.8 ± 0.02a</td>
<td>22.5 ± 0.04a</td>
</tr>
<tr>
<td>HHP-120</td>
<td>64.3 ± 0.22b</td>
<td>75.2 ± 0.22a</td>
<td>78.1 ± 0.15b</td>
<td>13.8 ± 0.06b</td>
<td>20.8 ± 0.23b</td>
</tr>
<tr>
<td>HHP-240</td>
<td>63.9 ± 0.08b</td>
<td>73.9 ± 0.62b</td>
<td>76.8 ± 0.90c</td>
<td>12.9 ± 0.12c</td>
<td>18.2 ± 0.18c</td>
</tr>
<tr>
<td>HHP-360</td>
<td>62.9 ± 0.34c</td>
<td>72.8 ± 0.13c</td>
<td>74.7 ± 0.13d</td>
<td>11.8 ± 0.23d</td>
<td>16.3 ± 0.11d</td>
</tr>
<tr>
<td>HHP-480</td>
<td>62.6 ± 0.21c</td>
<td>70.8 ± 0.18d</td>
<td>73.2 ± 0.05e</td>
<td>10.6 ± 0.41e</td>
<td>11.4 ± 0.35e</td>
</tr>
<tr>
<td>HHP-600</td>
<td>61.6 ± 0.15d</td>
<td>69.5 ± 0.08e</td>
<td>71.4 ± 0.18f</td>
<td>9.8 ± 0.11e</td>
<td>8.6 ± 0.05f</td>
</tr>
</tbody>
</table>

Means of triplicate determination ± S.D. with the different letter in a column within each parameter are significantly different ($p < 0.05$). $T_g$: onset temperature; $T_p$: peak temperature; $T_m$: concluding temperature; $\Delta T$ ($T_m$ - $T_p$): gelatinization temperature range; $\Delta H$: transition enthalpy.

### Table 4

Pasting properties of native and HHP-modified starches.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Samples</th>
<th>Native</th>
<th>HHP-120</th>
<th>HHP-240</th>
<th>HHP-360</th>
<th>HHP-480</th>
<th>HHP-600</th>
</tr>
</thead>
<tbody>
<tr>
<td>PV (cP)</td>
<td></td>
<td>4019 ± 0.33a</td>
<td>3608 ± 0.21b</td>
<td>3263 ± 0.13c</td>
<td>3006 ± 0.18d</td>
<td>972 ± 0.21e</td>
<td>371 ± 0.05f</td>
</tr>
<tr>
<td>BD (cP)</td>
<td></td>
<td>1645 ± 0.21a</td>
<td>1578 ± 0.15b</td>
<td>1003 ± 0.62c</td>
<td>960 ± 0.12c</td>
<td>133 ± 0.02d</td>
<td>150 ± 0.01e</td>
</tr>
<tr>
<td>SB (cP)</td>
<td></td>
<td>1915 ± 0.01a</td>
<td>1781 ± 0.12b</td>
<td>1765 ± 0.06b</td>
<td>1744 ± 0.28c</td>
<td>418 ± 0.12d</td>
<td>347 ± 0.23e</td>
</tr>
<tr>
<td>PV (cP)</td>
<td></td>
<td>4293 ± 0.22a</td>
<td>4211 ± 0.21a</td>
<td>4004 ± 0.05b</td>
<td>3811 ± 0.06c</td>
<td>1257 ± 0.09d</td>
<td>568 ± 0.28e</td>
</tr>
<tr>
<td>PT (C)</td>
<td></td>
<td>63.7 ± 0.15d</td>
<td>63.6 ± 0.22d</td>
<td>64.5 ± 0.11d</td>
<td>65.4 ± 0.14c</td>
<td>67.4 ± 0.13b</td>
<td>68.8 ± 0.11a</td>
</tr>
<tr>
<td>Pt (min)</td>
<td></td>
<td>4.26 ± 0.25e</td>
<td>4.67 ± 0.09d</td>
<td>4.86 ± 0.11c</td>
<td>4.93 ± 0.15c</td>
<td>5.26 ± 0.18b</td>
<td>5.73 ± 0.32a</td>
</tr>
</tbody>
</table>

Means of triplicate determination ± S.D. with the different letter in a row within each property are significantly different ($p < 0.05$). “cP” is the rapid viscosity units. PV: peak viscosity; BD: breakdown viscosity; FV: final viscosity; SB: setback viscosity; PT: pasting temperature, Pt: peak time.
attributed to the intact starch granule structure retained after HHP treatment; these granules had reduced the susceptibility to amyloytic enzymes (Blaszczak, Valverde, et al., 2005). Another reason for the increase in SDS content might be the smaller volume of amylose–amylopectin complexes formed in starch granules as a result of HHP (Lulien-Pellerin & Balny, 2002).

Normally, RS formation depends on food processing, temperature, starch gelatinization, storage conditions, and starch retrogradation. RS protects against colorectal cancer, controls serum cholesterol levels, decreases the glycemic index, and reduces insulinemic responses (Asp, Van Amelsvoort, & Hautvast, 1996). The increase in RS content of HHP-modified CBS reflected that strong amylose–amylose and amylose–amylopectin interactions occurred during treatment. Similar findings have been reported in amaranth, quinoa, and wheat starches (Linsberger-Martin, Lukasch, & Berghofer, 2012). Therefore, HHP treatment reduced the hydrolysis of CBS and improved its potential health benefits by lowering RDS content and increasing SDS and RS levels.

4. Conclusions

At 120–480 MPa, HHP treatment did not affect the ‘A’-type crystalline pattern of CBS, while at 600 MPa, HHP resulted in a similar ‘B’-type pattern. Modified CBS granules had more rough surfaces, higher AMC, and lower RC. Additionally, HHP modification decreased hardness, swelling power, and viscosity and increased pasting temperature and thermostability of CBS. Compared with native starch, HHP-modified CBS had lower in vitro hydrolysis, lower RDS content, and higher SDS and RS levels. The in vitro digestibility, physicochemical, and textural properties of CBS were pressure dependent. Therefore, HHP treatment is an efficient non-thermal modification method for improving the in vitro digestibility, physicochemical, and textural properties of CBS.

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