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Author contributions

Rongbin Cui: Methodology, Investigation, Writing – original draft, Writing - Review & Editing. **Michelle Ji Yeon Yoo**: Writing – Review & Editing. **Fan Zhu:** Conceptualization, Methodology, Writing – Review & Editing, Supervision, Project administration.

Purple sweetpotato flour (PSPF)

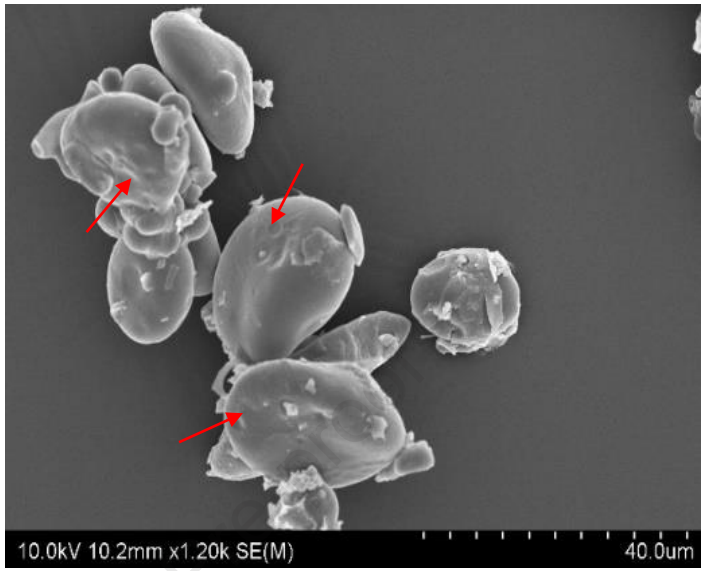
Wheat flour (WF)

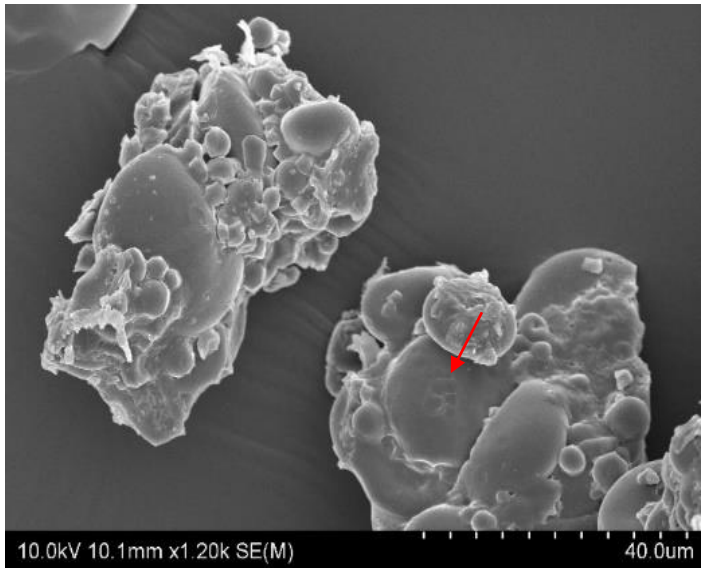
Microwave treatment (MWT)

VS

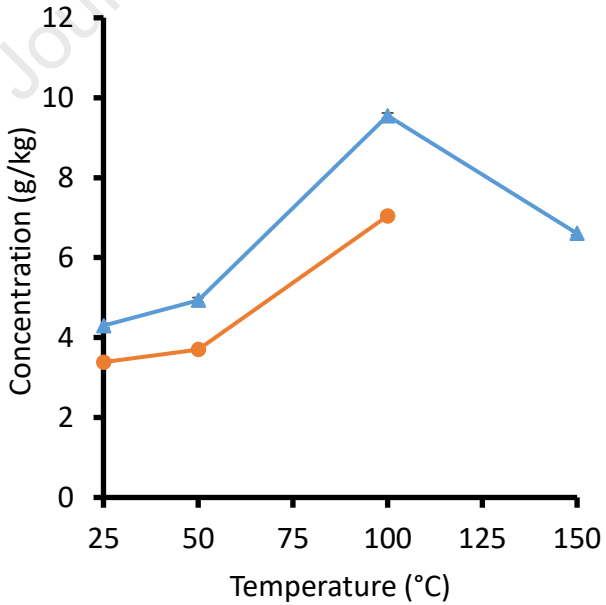
Conventional heat treatment (CHT)

Microstructure of WF after CHT at 50 °C

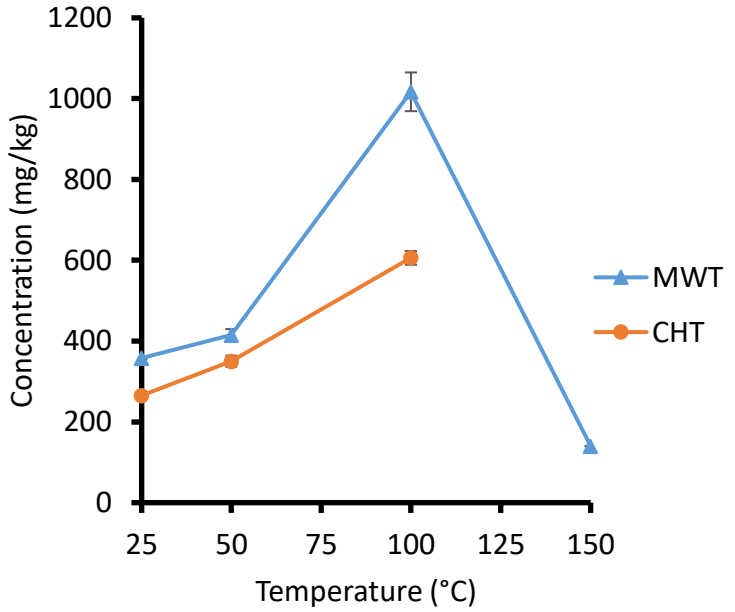




Total phenolic acids in PSPF after MWT



Total anthocyanins in PSPF after MWT



Comparison of microwave and conventional heating on physicochemical properties and phenolic profiles of purple sweetpotato and wheat flours

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Abstract

The hydrothermal treatment assisted with microwaves on physicochemical properties and phenolic profiles of purple sweetpotato flour and wheat flour was studied and compared with those of conventional heat treatment. Both treatments significantly decreased the enthalpy change, pasting viscosity and gelling capacity of flours, while increasing particle size, gelatinization temperatures and content of resistant starch. Microwaves induced more severe damages on the starch granule morphology and crystallinity than conventional heat treatment, which could be attributed to the intensive friction and collision produced by electromagnetic waves. The total concentrations of hydroxycinnamic acid derivatives and anthocyanins in purple sweetpotato flour extracted using microwaves at 100 °C were 36% and 68% higher than those in the samples after conventional heat treatment at 100 °C, respectively. Electromagnetic waves caused a higher extent of cell damage due to sudden temperature rise during microwave treatment. Overall, microwave treatment has potential to produce novel functionalities and nutritional values of flours and can be applied as an effective approach for other starch-based food products.

Keywords: *Ipomoea batatas*; microwave heating; conventional heating; dielectric property; phenolics; resistant starch

1. Introduction

Microwaves are electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz. In food industry and research, microwaves with a frequency of 2.45 GHz are most commonly used (Jiang, Liu, & Wang, 2018). The electric field of microwave radiation vibrates at high frequency, leading to the continuous orientation of polar and ionizable molecules (e.g., water and mineral salts). The altered molecules rub together and collapse with surrounding molecules through electromagnetic induction, generating heat (Tao et al., 2020). The effect of microwave treatment (MWT) on a food material depends on its dielectric properties. This term is mainly characterized by dielectric constant (ε', refers to the ability of a material to store energy at an electric field) and loss factor (ε'', refers to the ability of a material to convert electromagnetic energy to heat energy) (Jiang et al., 2018). Dielectric properties are influenced by several factors such as food composition, microwave frequency, heating temperature, and treatment duration (Tao et al., 2020). Thermal properties of food are also affected by microwaves. For example, MWT was used for starch modification with improved thermal stability and decreased retrogradation tendencies (Oyeyinka, Umaru, Olatunde, & Joseph, 2019).

MWT has been widely used in food processing such as extraction, dehydration, thawing, and pasteurization (Jiang et al., 2018). It is gradually replacing some conventional heat treatments (CHT) due to its shorter processing time, greater penetration depth, higher efficiency and potentially better quality of products, even though it still needs to be further studied and optimized in relation to experimental conditions (Guo, Sun, Cheng, & Han, 2017). Starch modification can be achieved by MWT due to its homogenous heating throughout the entire sample and faster heating rate than conventional heating (Bilbao-Sáinz, Butler, Weaver, & Bent, 2007). The structural changes of starch from microwave-delivered energy lead to changes in starch gelatinization, pasting viscosity, and digestibility (Zeng et al., 2016; Oyeyinka et al., 2019). The inactivation of polyphenol oxidase by microwave heating could solve the problem

of discoloration in fruit-based products (Cavalcante, Funcia, & Gut, 2021). Moreover, MWT under high energy (≥ 500 W) tended to have better effectiveness and lower manufacturing cost in food processing than non-thermal processing techniques such as high-pressure processing and ultrasonication (Li et al., 2021). These advantages make MWT an attractive technique for food industries. Therefore, the application of microwaves to modify food structures and functionalities should be extended to include a range of staple foods such as starch based food systems.

Sweetpotato (*Ipomoea batatas*) is an important staple crop in many countries (Wang, Nie, & Zhu, 2016). Starch is the major component in sweetpotato root and is critical to determine its special physicochemical properties and valued functionalities. Purple-fleshed sweetpotato varieties contain a range of bioactive compounds such as polyphenols, dietary fibers, minerals, and vitamins (Cui & Zhu, 2019). In particular, they are rich in a range of different polyphenols including hydroxycinnamic acid derivatives (HADs) and anthocyanins. These polyphenols have positive health effects on humans including antioxidant capacity, antiangiogenesis and hepatoprotection (Wang et al., 2016). While sweetpotato is normally eaten in cooked form, the dried roots can be used in the production of flour to extend its application range and shelf life (Trancoso-Reyes et al., 2016). Because of the similar processing properties as cereals, sweetpotato flour can partially/completely swap cereal flour (e.g., wheat flour) for the manufacture of food products (e.g., gluten free products and steamed bread) with enhanced nutritional values (Wang et al., 2016; Zhu & Sun, 2019). For value-added processing, sweetpotato flour remains to be subjected to a range of modifications to expand the range of functionalities for different food applications. Previous studies showed that flour prepared from microwave blanched sweetpotato had higher sensory acceptability than steamed flour (Dawkins & Lu, 1991). This may be partially attributed to the differences in bioactives compositions (Lu et al., 2010). More anthocyanins and β-carotene in microwave blanched flour

may be an attractive attribute for consumers (Guo et al., 2017). However, there has been no systematic report on the phenolic profiles and physicochemical aspects of microwave-treated sweetpotato flour.

The objective of this study was to compare the effects of MWT and CHT under various heating temperatures (up to 150 °C) on the physicochemical properties of PSPF, including microstructure, *in vitro* starch digestibility, thermal and pasting properties, gel texture, *in vitro* antioxidant potential and phenolic profiles. Wheat flour (WF), as a well-studied food product, was employed for comparison purpose to investigate the food composition effect of MWT. It was hypothesized that the extent of change for flour functionalities under MWT would be greater than that of the changes under CHT due to the presence of electromagnetic waves. The results of this study will be important to further develop microwave treated flours for novel applications in a green and efficient way.

2. Materials and methods

2.1. Materials

The purple-fleshed sweetpotato variety (Purple Dawn) was cultivated in Ruawai, Northland, New Zealand. The crop was grown on the alluvial plains under normal agronomic practices in 2020. Around 10 kg of sweetpotato roots were randomly selected at the harvesting time. The roots were manually washed, cut into small cubes and freeze-dried (FreeZone Plus, Labconco, Kansas City, MO, USA). Purple sweetpotato flour (PSPF) was obtained by blending the dried roots using a Kenwood speed blender (Havant, UK) and then passed through a 0.05 mm mesh sieve. Commercial wheat flour (WF) was purchased from Countdown supermarket (Auckland, New Zealand). Flours were stored in a desiccator at room temperature (~23 °C) in the dark until use. The nutritional composition of the flours is summarized in Table 1.

Porcine pancreatic α-amylase (P1750), invertase (I4504), Folin-Ciocalteau reagent, gallic acid, Trolox, chlorogenic acid and cyanidin-3-glucoside were obtained from Sigma-Aldrich Chemical Ltd. (St Louis, USA). Amyloglucosidase (E-AMGDF) was obtained from Megazyme (Wicklow, Ireland). All other chemicals and reagents used were of analytical grade.

2.2. Hydrothermal treatment assisted with microwave (MWT)

Flour suspensions were prepared at a flour-to-water ratio of 1:10 (w/v) in Teflon-coated vessels. MWT at 2.45 GHz was performed using an advanced flexible microwave synthesis reactor from Milestone (Sorisole, Italy) with a power density range between 0 and 90 W/g (Supplementary Table 1). The flour suspensions were treated at 25, 50, 100 and 150 °C for 15 min. The heating temperature and time were chosen based on our preliminary trial and conditions for possible food applications. To control the temperature increase, an incident power was applied to the system to reach 25 and 50 °C within 1 min ramp time, whereas a ramp time of 4 min was set up for the system to reach 100 and 150 °C (Supplementary Fig. 1). Sample temperature was measured on real time by a fiber optic temperature sensor (Milestone, Sorisole, Italy). A magnetic stirring bar was built in the system, allowing consistent mixing during treatment. After MWT, the flour suspensions were cooled down at ambient condition before freeze drying.

2.3. Conventional heat treatment (CHT)

Flour samples (20 g, dry basis (db)) with 200 mL water were placed in a 250 mL beaker. CHT was conducted by heating the flour suspension on a hot plate magnetic stirrer (MR 3001, Heidolph Instruments, Germany). Once the temperatures of the flour suspensions reached 25, 50 and 100 °C, they were continuously heated for 15 min. The time for flour suspensions to reach 25, 50 and 100 °C was measured as 1.5, 5 and 12 min, respectively (Supplementary Fig. 2). The flour suspension was continuously stirred by a magnetic stirring bar at 250 rpm to ensure uniform heat treatment. The hot plate was turned off after 15 min, but with continuous

stirring to match with the remaining time profile in MWT. After CHT, the flour suspension was cooled down at ambient condition and freeze dried.

2.4. Scanning electron microscopy (SEM)

A Hitachi SU 70 scanning electron microscopy (Tokyo, Japan) was used to analyze the morphological changes in the flours (Cui & Zhu, 2020). Details of the methods can be found in the Supplementary Materials.

2.5. Particle size analysis

The particle size distribution of flours was conducted using a Mastersizer 2000 particle size analyzer (Malvern Instruments, Worcestershire, UK) (Cui & Zhu, 2020). Details of the methods can be found in the Supplementary Materials.

2.6. Fourier transform infrared spectroscopy (FTIR)

The spectra of the flours were recorded by a Bruker Vertex 70 FTIR spectrometer equipped with a single bounce diamond accessory (Bruker Daltonics, Bremen, Germany) using the method of Cui and Zhu (2020). Details of the methods can be found in the Supplementary Materials.

2.7. *In vitro* starch digestibility

The *in vitro* starch digestibility was determined according to the method reported by Englyst, Kingman, and Cummings (1992) with modifications. Details of the methods can be found in the Supplementary Materials.

2.8. Gelatinization properties

Gelatinization properties of flours were assessed by a Q1000 differential scanning calorimetry (DSC) (TA Instrument, New Castle, USA). Details of the methods can be found in the Supplementary Materials.

2.9. α-Amylase activity and pasting analysis

The α-amylase activity was measured using the α-amylase activity assay kit (Megazyme, Wicklow, Ireland). The pasting properties of flours were analyzed by a Physica MCR 301 rheometer (Anton Paar, Graz, Austria) followed the method of Cui and Zhu (2020). Details of the methods can be found in the Supplementary Materials.

2.10. Gel textural properties

The flour paste after the pasting analysis was stored at 4 °C for 24 h before texture profile analysis (TPA) as described by Cui and Zhu (2020). Details of the methods can be found in the Supplementary Materials.

2.11. Color

The color properties (*L**, *a**, and *b**) of flours were determined with a CR-300 Chroma-meter (Minolta Camera Co., Osaka, Japan) according to the method of Kumar, Sadiq, and Anal (2020). Details of the methods and related results and discussion can be found in the Supplementary Materials.

2.12. Preparation of methanolic extract of flour samples

The methanolic extracts of flour samples were prepared based on the method of Inglett, Rose, Chen, Stevenson, and Biswas (2010). Details of the methods can be found in the Supplementary Materials.

2.13. Total phenolic content (TPC) and *in vitro* antioxidant potential

TPC was determined according to the Folin-Ciocalteau method described by Inglett et al. (2010), using gallic acid as standard. The results were expressed as g gallic acid equivalents (GAE) per kg db. *In vitro* antioxidant potential was measured using the FRAP assay based on the method of Xu, Chen, Cao, Xia, and Jiang (2016). The results of *in vitro* antioxidant potential

were expressed as mmol Trolox equivalents (TE) per kg db. Details of the methods and related results and discussion can be found in the Supplementary Materials.

2.14. Determination of composition of individual polyphenol compounds

The identification of phenolic acids and anthocyanins and their quantification were done by hybrid quadrupole time-of-flight (MicrOToF) mass spectrometry (Bruker Daltonics, Bremen, Germany) and high performance liquid chromatography coupled with diode array detector (HPLC-DAD) (Agilent Technologies, Wilmington, DE, USA) based on the method used in a previous report with minor modifications (Cui & Zhu, 2019). The HPLC conditions and quantification methods can be found in the Supplementary Materials. The concentrations of phenolic acids and anthocyanins were expressed as g chlorogenic acid equivalents (CAE)/kg db and mg cyanidin 3-glucoside equivalents (CGE)/kg db, respectively.

2.15. Statistical analysis

All experiments were performed in triplicate (n = 3), and data were presented as mean ± standard deviation (SD). One-way analysis of variance (ANOVA) with Duncan's multiple comparison tests were performed to analyze the significant differences ($p < 0.05$). These analyses were computed with SPSS 25.0 software (IBM Corp., Armonk, NY, USA).

3. Results and discussion

3.1. Morphological characterization of microwave and conventional heat treated flours

PSPF and WF had shown a significant variation in microstructure and they varied in the sizes and shapes (Fig. 1). The starch granules in the PSPF showed oval and polygonal shapes with smooth surface, which was in agreement with Trancoso-Reyes et al. (2016) who reported the same type of sweetpotato starch morphology. Some non-starch components (e.g., pectins) were infused around the granules. Wheat starch granules were embedded in the protein matrix. Two

187 distinct types of starch granules were observed in WF. They were the large and discoid shape
188 of A-type starch and the small and spherical shape of B-type starch, which was in accordance
189 with the report by Bilbao-Sáinz et al. (2007).

190 MWT altered the starch granule morphology, and the extents of the changes varied with
191 microwave temperatures (Fig. 1). Starch granule integrity of PSPF was maintained after MWT
192 at 50 °C, whereas significant changes were observed after MWT at higher temperatures (100
193 and 150 °C). After MWT at 25 °C, there were some dents and roughness on the surface of
194 wheat starch granules. With a higher microwave temperature, the surface depression of the
195 wheat starch became more obvious. Both flours treated by microwave showed aggregated
196 microstructure with irregular surfaces after gelatinization.

197 The sweetpotato starch was relatively intact after CHT at 25 (Fig. 1, E) and 50 °C (Fig. 1, F),
198 whereas slight dents were observed on wheat starch after CHT at 50 °C (Fig. 1, M). The heating
199 method had important effects on the microstructure, especially at low temperature (< 100 °C).
200 For example, WF treated by CHT at 25 °C showed relatively smooth granule surfaces. WF
201 treated by MWT at 50 °C had more damaged starch granules compared with that by CHT (Fig
202 1, I *vs* M). Microwave energy could generate heat rapidly and penetrate the medium of starch
203 granules by molecular frictions, leading to greater damage on starch granules than CHT (Tao
204 et al., 2020).

205 Under the same temperature, starch in WF was damaged more severely than that of starch in
206 PSPF (e.g., Fig. 1, I *vs* B). WF starch with a higher amylose content may be more susceptible
207 to hydrothermal treatment than PSPF starch, as amorphous part (amylose) of the starch
208 granules got affected more than the crystalline regions (Emami, Perera, Meda, & Tyler, 2012).

3.2. Particle size of microwave and conventional heat treated flours

PSPF and WF showed variations in the particle size (Table 2). For example, D [4, 3] of PSPF after CHT at 25 °C (183 μm) was larger than that of WF (155 μm). This could be largely attributed to the differences in morphological structure of starch granules and their chemical compositions (Fig. 1; Table 1).

The particle size of PSPF appeared to be similar to that of the samples after MWT at 25 and 50 °C (Table 2). This indicated that these microwave temperatures did not cause the agglomeration of the flour. Both flours showed a distinct bimodal particle size distribution at low temperatures (25 and 50 °C) (Supplementary Fig. 3). The particle size significantly increased with increasing microwave temperature at 100 °C for both flours due to gelatinization. However, the increase in particle size is different from the size of aggregates observed by SEM (Fig. 1). A significant part of the aggregates was not quantified by the light scattering method due to the excessive aggregation after starch gelatinization (Zhang, Zhang, Thakur, Zhang, & Wei, 2019).

Particle size of flours affected by CHT was similar to that affected by MWT up to 100 °C. However, the extents of changes in the particle sizes affected by MWT and CHT were different (Supplementary Fig. 4). For example, D [3, 2] of PSPF treated by MWT at 100 °C (99.7 μm) was higher than that treated by CHT at 100 °C (45.1 μm). The electromagnetic waves can transform more energy to heat, causing more extensive changes in particle size of flours (Tao et al., 2020).

MWT at 150 °C significantly decreased the particle size of PSPF, whereas the opposite trend was observed for WF. This could be due to the differences in starch structure and other components (e.g., protein) presented in the flours. Thermal degradation of starch may occur above 120 °C (Tao et al., 2020). As the MWT temperature increased, cross-linking of gluten

proteins and the formation of isopeptide bonds were promoted after treatment (Xiang, Zou, Liu, & Ruan, 2020). This led to more aggregation and increased particle size of WF after MWT at 150 °C.

3.3. FTIR of microwave and conventional heat treated flours

The differences in the peak positions between the spectra of PSPF and WF suggested variations in their structure and chemical compositions (Supplementary Fig. 5). For example, the amide II peak located at the band of 1600–1500 cm^{-1} was only observed in WF, which could be attributed to its higher protein content than PSPF (Xiang et al., 2020).

PSPF treated at different microwave temperatures showed similar peak positions of FTIR spectra (Supplementary Fig. 5). This indicated that MWT did not affect the chemical bonds of flours. Similar observations were reported for microwave treated potato and lycoris starches (Zhang et al., 2019; Kumar, Singh, Sharanagat, Patel, & Kumar, 2020). The fingerprint region of starch has typical bands of 1200–800 cm^{-1}. In particular, the ratio of bands at 1047/1022 cm^{-1} could reflect the ordered structure and hydrogen bond strength of starch (Kumar, Singh et al., 2020). The intensity ratios ($R_{1047/1022}$) of both flours were decreased with increasing microwave temperature (Table 3), which suggested that the two treatments disrupted the crystallinity of starch.

The intensity ratio of microwave treated flours was significantly lower than that of the conventional heat treated flours (Supplementary Fig. 6). This suggested that it had higher ability to affect starch molecules through microwave induced dielectric heating effect. The dielectric constant decreased with increasing temperature. This improved the absorption and conversion abilities of the starch system, leading to more local accumulation of heat (Tao et al., 2020).

The extents of changes in peak intensities induced by MWT and CHT also appeared differently between PSPF and WF. For example, the extents of microwave treated changes in protein structure (1700–1500 cm^{-1}) were more prominent in the WF than in the PSPF, which could be attributed to the higher protein content of the former. Again, the differences in intensity ratio suggested that WF was more susceptible to hydrothermal treatment than PSPF (Supplementary Fig. 6).

3.4. *In vitro* starch digestibility of microwave and conventional heat treated flours

Starch in PSPF was less susceptible to enzymatic hydrolysis than that of the WF (Table 2). This may be due to their different starch contents, which significantly affected the amount of glucose released during hydrolysis. The susceptibility to enzymatic hydrolysis was affected by the particle size of flours, and PSPF with larger particles showed lower susceptibility (Zhao et al., 2019).

The nutritional fractions of starch components in flours were much affected by MWT (Table 2). The contents of RDS and RS were increased by increasing microwave temperature (e.g., RDS of microwave-treated PSPF from 28.2% to 40.1% and RS from 13.4% to 19.8%), whereas that of the SDS content was decreased (e.g., from 35.6% to 17.3%). The increased starch digestibility after MWT was also reported in starches from normal, high-amylose, and waxy barleys (Emami et al., 2012). The pores and cracks induced by the MWT on the surface of starch granules promote access of enzymes into the granules and facilitate the chemical reactions (section 3.1). The reduction of RS (Type 2) after MWT at 50 °C could be attributed to the disruption of crystalline structure of starch induced by MWT (section 3.3). The increased RS content after MWT at 100 °C could be due to the amylose-amylose interactions and the retrogradation of starch promoting the formation of RS (Type 3) during cooling (Zeng et al., 2016).

The rapid heating and more significant starch degradation generated by MWT resulted in a more pronounced effect on the starch nutritional fractions (e.g., more RDS after MWT at 100 °C in PSPF) compared to CHT (Supplementary Fig. 7). This suggested the potential of MWT to improve the digestibility of food systems. It was reported that microwave treated rice starch had higher RDS content than conventional heat treated rice starch (Li, Han, Xu, Xiong, & Zhao, 2014).

The change of starch digestibility not only depends on the treatment conditions but also on the food composition. WF tended to be more sensitive to enzymatic hydrolysis after thermal treatment than PSPF partially due to their differences in the total starch content and granular structure (Emami et al., 2012). The phenolics in PSPF could reduce the enzymatic hydrolysis through interactions with starch and digestive enzymes (Zhu, 2015). The increased phenolics contents after MWT (Table 5) could further promote the formation of starch-phenolic complexes, affecting the starch digestibility (Zhao et al., 2019).

3.5. Gelatinization properties of microwave and conventional heat treated flours

The gelatinization temperatures (T_o, T_p, and T_c) of PSPF were higher than those of WF. For example, T_c of PSPF and WF after CHT at 25 °C were 73.5 and 67.9 °C, respectively (Table 3). The differences in the gelatinization temperatures could be due to the differences in the starch composition, structure and interactions between starch and other components. For example, starch can interact with cations like Na^+, Mg^{2+}, and Ca^{2+} to form complexes during gelatinization, inhibiting the starch-starch associations in the system (Gujral, Park, & Baik, 2008).

The gelatinization temperatures of flours increased with increasing microwave temperature, but the ΔH decreased. For example, T_o (60.2 °C) of PSPF after MWT at 50 °C was ~ 6 °C higher than that after MWT at 25 °C, while its ΔH (7.19 J/g) was lower than that after MWT

304 at 25 °C (7.84 J/g). The results were in agreement with previous studies on rice and maize
305 flours (Román, Martínez, Rosell, & Gómez, 2015; Villanueva, Harasym, Muñoz, & Ronda,
306 2018). The increase of gelatinization temperatures could be due to the enhancement of the
307 amylose-amylose or amylose-amylopectin interactions among the starch chains induced by the
308 treatment (Oyeyinka et al., 2019). The decrease in Δ*H* suggested the disruption of the ordered
309 lamellar arrangements within semicrystalline region of the starch granules during MWT
310 (Oyeyinka, Akinware, Bankole, Njobeh, & Kayitesi, 2021).

311 CHT produced a similar rearrangement in the structure of flour matrix, but the extent of
312 changes was lower than that of MWT (Supplementary Fig. 8). The molecular vibration of polar
313 molecules under MWT could accelerate the impact on gelatinization properties (Jiang et al.,
314 2018). The lower Δ*H* in microwaved samples than that of the conventional heated samples
315 suggested the less ordered structure of the remaining crystals (shown in section 3.3).

316 Both flours were completely gelatinized at temperatures of ≥ 100 °C, while WF seemed to be
317 more susceptible to MWT at low temperatures (25 and 50 °C) than PSPF. During the heating,
318 the pectin molecules in PSPF may form a barrier on starch granule surface. This could restrict
319 the water diffusion to the amorphous regions and reinforce the granule structure during thermal
320 treatment (Kumar, Sadiq et al., 2020). Furthermore, phenolic compounds may interact with
321 amylose or cellulose via hydrogen bonding, affecting the gelatinization properties of flours
322 (Zhu, 2015).

323 3.6. α-Amylase activity and pasting properties of microwave and conventional heat treated
324 flours

325 The viscosities of PSPF were much lower than those of WF during pasting event (Table 4).
326 Such differences could be largely due to the flour composition and starch properties. The
327 endogenous α-amylase could break starch into maltodextrins and glucose, which consequently

328 reduced the viscosity of flour paste (Hagenimana, Vezina, & Simard, 1992). FV of PSPF was
329 lower than that of WF, this could be due to lower amylose content and higher amount of dietary
330 fiber/polyphenols of the former. The amylose can interact with dietary fiber and polyphenols
331 by hydrogen bonding, which greatly restricts the amylose-amylose interaction, resulting in the
332 lower FV of PSPF (Trancoso-Reyes et al., 2016).

333 α-Amylase lost its activity upon heating at 40 °C and it can be inactivated at 100 °C due to the
334 denaturation of protein (as shown in section 3.3) (Supplementary Fig. 5) (Hagenimana et al.,
335 1992). MWT significantly decreased the viscosities of PSPF and WF (Table 4). For example,
336 PSPF treated by MWT at 50 °C had lower PV (1.88 Pa·s) than that by MWT at 25 °C (2.35
337 Pa·s). WF treated by MWT at 50 °C showed much lower HPV (2.07 Pa·s) and FV (4.68 Pa·s)
338 than that after MWT at 25 °C (HPV, 2.23 Pa·s; FV, 5.35 Pa·s). This could be largely attributed
339 to the disruption of starch granule structure, which reduced the water absorption and binding
340 capacity. The decrease in viscosities of the flours was consistent with the results of microwave-
341 treated starches from cassava, lycoris and Bambara groundnut (Oyeyinka et al., 2019; Zhang
342 et al., 2019; Oyeyinka et al., 2021). Microwave also affected the BD and SB in the same manner.
343 For example, PSPF treated by MWT at 50 °C had much lower BD (1.70 Pa·s) than that after
344 MWT at 25 °C (2.17 Pa·s). The reduced BD and SB suggest the enhanced stability of flours
345 delayed retrogradation from the MWT (Villanueva et al., 2018).

346 MWT could be more effective to inactivate α-amylase in PSPF than CHT due to more efficient
347 penetration of microwave power (Supplementary Fig. 9). In general, the changes in viscosities
348 of flours treated by MWT were more pronounced than those of CHT (Supplementary Fig. 9).
349 These results were in agreement with a previous study showing higher reduction of viscosity
350 by MWT than by steam treatment (Trancoso-Reyes et al., 2016). During the microwave process,
351 polar components (such as water) vibrated rapidly, the friction and collision between water

molecules and starch granules increased the temperature in a short time. The forces brought physical damages to the granules, and high level of disruption to the paste.

Under MWT and CHT, the extents of viscosity reduction in PSPF were greater than those of WF. Such differences could be due to the presence of α-amylase in PSPF. Another possible explanation was that the pasting viscosity of starch with low amylose content was more likely to be influenced by microwave than high amylose starch (Emami et al., 2012).

3.7. Gel textural properties of microwave and conventional heat treated flours

The gels of PSPF showed lower hardness and adhesiveness than those of WF (Table 4). The presence of α-amylase in PSPF significantly decreased its gelation (Zhu & Sun, 2019). The lower amylose content and higher pectin/phenolic contents in PSPF further contributed to the weaker gelation compared to WF (Zhu, 2015).

Textural properties of both flour gels were much affected by MWT. For example, the hardness (0.209 N) and adhesiveness (0.811 N·s) of WF treated by MWT at 50 °C were much lower than those after MWT at 25 °C (hardness, 0.348 N; adhesiveness, 1.313 N·s). These results were consistent with those reported by Zhang et al. (2019), who showed that lycoris starch could not form a rigid structure after MWT. The swelling of starch granules and breakdown of amylopectin structure at elevated temperatures weakened the hardness and adhesiveness of flours after MWT (Zhang et al., 2019).

The gels formed after MWT were much weaker than those formed after CHT (Supplementary Fig. 10). The higher reduction in hardness could be due to more damage on the starch induced by microwave compared to that of starch in conventional heat treated flours. The α-amylase activity and pectin/phenolic contents of PSPF were significantly higher than those of WF. The effect of gel weakening was more prominent for PSPF than for WF. These trends in gel texture properties were consistent with the changes found in pasting viscosities from section 3.6.

3.8. Phenolic acids of microwave and conventional heat treated PSPF

Major phenolic acids in PSPF were identified as hydroxycinnamic acid derivatives (HADs). A total of 10 HADs were detected from the extract of PSPF (Table 6; Supplementary Fig. 12; the profile of PSPF treated after MWT at 100 °C is given as an example). The results were in agreement with those of previous studies on PSPF (Cui & Zhu, 2019). 3-5-Di-CQA was the dominant HAD in all of the samples, followed by 5-CQA, while CA was the least abundant HAD.

The HAD profile of the extracts did not change with MWT temperatures, whereas the concentration of HADs increased as the temperature increased to 100 °C. A sharp increase in the concentration of total HADs from 4.93 to 9.55 g CAE/kg db was obtained when the microwave temperature increased from 50 to 100 °C. Similar trends were obtained for the individual HADs with different microwave temperatures. The increase of temperature may facilitate the release of phenolic acids due to the rupture in the plant cell walls (Xu et al., 2016). The decreased activity of various oxidase enzymes (e.g., polyphenol oxidase (PPO), peroxidase) under high temperature treatment may also prevent the oxidation loss of phenolic acids (Jiang et al., 2018). The decrease in the concentrations of HADs was observed after MWT at 150 °C except for 3-CQA and 4-CQA. HADs are polar compounds, which can absorb microwave energy directly and the degradation may occur under intensive microwave conditions (Liazid, Palma, Brigui, & Barroso, 2007). Indeed, phenolics with fewer substituents in the aromatic ring tended to have higher stability during the MWT (Liazid et al., 2007). The concentrations of ferulic acid from wheat bran, whole WF, and refined WF were increased after MWT 100 °C, especially in the bran fraction (Lu & Luthria, 2016). MWT improved the extractability of phenolic compounds from WF due to the disruption of the cell wall components of endosperm and bran layers (Lu & Luthria, 2016).

The total concentration of HADs in the samples after MWT was higher than that in those after CHT (Supplementary Fig. 13). However, the extraction efficiencies of individual HAD in the MWT and CHT extracts were different. For example, the concentrations of CA (0.18 g CAE/kg db) and FQA (0.15 g CAE/kg db) extracted after MWT at 50 °C were lower than those extracted under CHT at 50 °C (CA, 0.28 g CAE/kg db; FQA, 0.17 g CAE/kg db). These results supported the findings of Galan et al. (2017) and suggested that the enhanced extraction of phenolic acids by MWT is likely through the rapid volumetric heating. The direction of heat transfer is from the "inside out" rather than from "outside in" (Jiang et al., 2018). The plant matrix could also be selectively heated, producing the changes in the chemical potential between phases with a higher yield of phenolic compounds (Seoane et al., 2017).

3.9. Anthocyanins of microwave and conventional heat treated PSPF

A total of 7 anthocyanins were identified in PSPF (Table 7; Supplementary Fig. 14; the profile of PSPF treated after MWT at 100 °C is given as an example). Peonidin 3-(6"-caffeoyl-6"'-*p*-hydroxybenzoylsophoroside)-5-glucoside was the major anthocyanin. The composition of anthocyanins was in agreement with the results of a previous study (Cui & Zhu, 2019).

The concentration of anthocyanins in PSPF increased with increasing MWT temperatures. The highest total anthocyanin concentration was achieved after MWT at 100 °C (1017 mg CGE/kg db). This is largely attributed to the cell wall disruption and PPO inactivation at this temperature (Cavalcante et al., 2021). However, MWT at 150 °C led to a reduction in the concentration of anthocyanins in PSPF. Similar observation in the concentration of anthocyanins was previously reported for bilberry after MWT at 150 °C for 10 min (Yue & Xu, 2008). The degradation of anthocyanins could be due to the formation of chalcone in the process of extreme thermal exposure or loss of glycosyl moieties (Xu et al., 2016).

MWT induced greater increases in the concentrations of individual and total anthocyanins than CHT (Supplementary Fig. 15). For example, the total concentration of anthocyanins in the PSPF after MWT at 100 °C was 68% higher than that after CHT at 100 °C. Peonidin 3-(6"-feruloylsophoroside)-5-glucoside was not detected in the sample after CHT at 50 °C, while it was detectable in the sample after MWT at 50 °C. These results agreed with a previous study on grape skins (Liazid, Guerrero, Cantos, Palma, & Barroso, 2011). Microwave-assisted extraction has a beneficial effect on the extraction process when compared to conventional extraction methods. This could be partially explained by the deeper disruption of the outer layer of cell walls by microwave leading to the release of more anthocyanins. In addition, the uniform heating and strong penetration power of microwave could be more effective to inactivate the PPO in PSPF (Cavalcante et al., 2021).

3.10. Potential use of MWT and significance of the study: A general discussion

This study demonstrated that MWT up to 90 W/g significantly affected the physicochemical properties of PSPF and WF as described in the sections above. MWT may be utilized for several food applications based on our results. The microwave treated flours had improved nutritional qualities of starch (e.g., enhanced RS content) which can be beneficial to patients suffering from high levels of serum cholesterol or other diseases (Table 2) (Zhang et al., 2019). The gelatinized flours after MWT at 100 °C can be used in ready-to-eat product formulations or as gelling agents (Table 3). MWT altered the rheological properties of flours (e.g., reduced BD), suggesting an enhanced stability of dough viscosity during heating (Table 4). This could allow more air to be retained in dough during bread baking for better development (Villanueva, Harasym, Muñoz, & Ronda, 2019). The microwave treated PSPF could also be a suitable ingredient for gluten-free bakery formulations due to the lack of gluten-type protein. MWT could facilitate the extraction of phenolic compounds (Tables 6 and 7). The increased extractability of phenolic acids and anthocyanins in PSPF are closely related with antioxidant

potential and plays a role for the reduction of starch digestibility through the interactions with starch and digestive enzymes (Zhu, 2015). However, excessive electromagnetic waves increased electrical energy absorbed into the solution and sample. This caused an increase in the motion of the polar molecules and generated extreme temperature, promoting the degradation of phenolic compounds. Response surface methodology could be used to optimize the extraction conditions for phenolic acids and anthocyanins in PSPF (Ahmed, Akter, $ Eun, 2011). The microwave heating technology for pasteurization and sterilization contributed to effectively destruction of pathogenic microorganisms (Guo et al., 2017). Therefore, foods incorporated with microwave treated flours could potentially have an improved shelf life. The microbiological studies related to the microwave treated flours remained to be studied.

Microwave technology has been applied to different food systems to improve the functionalities for specific applications (Inglett et al., 2010; Román et al., 2015; Kumar, Singh et al., 2020). However, these studies did no comparison between MWT and CHT. The differences in the analytical methods among different reports make the comparison of the results hardly possible. The direct comparison in the results between MWT and CHT on the flour properties at the same temperature was performed under the same experimental conditions in this study. The extents of changes in various physicochemical properties were not only dependent on the treatment conditions but also the types of flours. For example, non-starch components (e.g., dietary fiber and phenolic compounds) of PSPF significantly affected the gelatinization and pasting properties of PSPF during hydrothermal treatment. Within the same temperature, MWT increased the extractability of phenolic compounds in PSPF to a larger extent than CHT (Fig. 2). Because microwave can heat up foods using the energy of oscillating electromagnetic wave, it is possible to do selective and quick cooking (Galan et al., 2017). Another important parameter for the efficiency of MWT is the moisture content of the sample (Tao et al., 2020). Systems with high moisture content (> 40%) can quickly convert

microwave electromagnetic energy into heat energy (Tao et al., 2020). The physicochemical properties of PSPF and WF with high moisture content treated by microwave should be analyzed in the future.

4. Conclusions

The hydrothermal treatment assisted with microwaves significantly affected the structural and physicochemical properties as well as the phenolic profiles of PSPF and WF in a temperature-dependent manner. The starch in PSPF was less susceptible to MWT at low temperatures (25 and 50 °C) than the starch in WF as revealed by SEM. Particle size of flours was increased after MWT at 100 °C due to the aggregation of gelatinized starch. The chemical bonds of flours were not affected by MWT as measured by FTIR. The MWT at > 50 °C significantly increased the content of RS due to the increased starch retrogradation. Gelatinization temperatures increased with increasing microwave temperature, but viscosities and gelation decreased. Although CHT produced a similar change of flour functionalities, the extent of changes was lower than that of MWT. The electromagnetic waves from microwaves produced rapid heating and more significant starch degradation. Furthermore, MWT significantly improved the extractability of phenolic compounds from PSPF compared to that of CHT. However, excessive electromagnetic waves can cause phenolics degradation. These results proved the efficiency of MWT in the modifications of functional and nutritional properties of starch based food systems that took place in significantly shorter periods than CHT.

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Conflicts of interest

None.

References

Ahmed, M., Akter, M. S., & Eun, J. B. (2011). Optimization conditions for anthocyanin and phenolic content extraction form purple sweet potato using response surface methodology. *International Journal of Food Sciences and Nutrition*, *62*, 91–96. https://doi.org/10.3109/09637486.2010.511167.

Bilbao-Sáinz, C., Butler, M., Weaver, T., & Bent, J. (2007). Wheat starch gelatinization under microwave irradiation and conduction heating. *Carbohydrate Polymers*, *69*, 224–232. https://doi.org/10.1016/j.carbpol.2006.09.026.

Cavalcante, T. A. B. B, Funcia, E. S. M., & Gut, J. A. W. (2021). Inactivation of polyphenol oxidase by microwave and conventional heating: Investigation of thermal and non-thermal effects of focused microwaves. *Food Chemistry, 340,* 127911. https://doi.org/10.1016/j.foodchem.2020.127911.

Cui, R., & Zhu, F. (2019). Physicochemical properties and bioactive compounds of different varieties of sweetpotato flour treated with high hydrostatic pressure. *Food Chemistry, 299*, 125129. https://doi.org/10.1016/j.foodchem.2019.125129.

Cui, R., & Zhu, F. (2020). Effect of ultrasound on structural and physicochemical properties of sweetpotato and wheat flours. *Ultrasonics Sonochemistry*, *66*, 105118. https://doi.org/10.1016/j.ultsonch.2020.105118.

520 Dawkins, N. L., & Lu, J. Y. (1991). Physico-chemical properties and acceptability of flour
521 prepared from microwave blanched sweet potatoes. *Journal of Food Processing and*
522 *Preservation, 15*, 115–124. https://doi.org/10.1111/j.1745-4549.1991.tb00159.x.

523 Emami, S., Perera, A., Meda, V., & Tyler, R. T. (2012). Effect of microwave treatment on
524 starch digestibility and physico-chemical properties of three barley types. *Food and*
525 *Bioprocess Technology, 5*, 2266–2274. https://doi.org/10.1007/s11947-011-0688-2.

526 Englyst, H. N., Kingman, S., & Cummings, J. (1992). Classification and measurement of
527 nutritionally important starch fractions. *European Journal of Clinical Nutrition, 46*,
528 S33–S50. ISSN: 09543007.

529 Galan, A. M., Calinescu, I., Trifan, A., Winkworth-Smith, C., Calvo-Carrascal, M., Dodds, C.,
530 & Binner, E. (2017). New insights into the role of selective and volumetric heating
531 during microwave extraction: Investigation of the extraction of polyphenolic
532 compounds from sea buckthorn leaves using microwave-assisted extraction and
533 conventional solvent extraction. *Chemical Engineering and Processing: Process*
534 *Intensification, 116*, 29–39. https://doi.org/10.1016/j.cep.2017.03.006.

535 Gujral, H. S., Park, S. J., & Baik, B. K. (2008). Effect of added minerals on pasting of partial
536 waxy wheat flour and starch and on noodle making properties. *Cereal Chemistry, 85*,
537 97–101. https://doi.org/10.1016/j.foodchem.2013.01.085.

538 Guo, Q., Sun, D. W., Cheng, J. H., & Han, Z. (2017). Microwave processing techniques and
539 their recent applications in the food industry. *Trends in Food Science & Technology,*
540 *67*, 236–247. https://doi.org/10.1016/j.tifs.2017.07.007.

541 Hagenimana, V., Vezina, L. P., & Simard, R. E. (1992). Distribution of amylases within sweet
542 potato (*Ipomoea batatas* L.) root tissue. *Journal of Agricultural and Food Chemistry,*
543 *40*, 1777–1783. https://doi.org/10.1021/jf00022a010.

544 Inglett, G. E., Rose, D. J., Chen, D., Stevenson, D. G., & Biswas, A. (2010). Phenolic content
545 and antioxidant activity of extracts from whole buckwheat (*Fagopyrum esculentum*
546 Möench) with or without microwave irradiation. *Food Chemistry, 119*, 1216–1219.
547 https://doi.org/10.1016/j.foodchem.2009.07.041.
548 Jiang, H., Liu, Z., & Wang, S. (2018). Microwave processing: Effects and impacts on food
549 components. *Critical Reviews in Food Science and Nutrition, 58*, 2476–2489.
550 https://doi.org/10.1080/10408398.2017.1319322.
551 Kumar, S. R., Sadiq, M. B., & Anal, A. K. (2020). Comparative study of physicochemical and
552 functional properties of pan and microwave cooked underutilized millets (proso and
553 little). *LWT-Food Science and Technology, 128*, 109465.
554 https://doi.org/10.1016/j.lwt.2020.109465.
555 Kumar, Y., Singh, L., Sharanagat, V. S., Patel, A., & Kumar, K. (2020). Effect of microwave
556 treatment (low power and varying time) on potato starch: Microstructure, thermo-
557 functional, pasting and rheological properties. *International Journal of Biological*
558 *Macromolecules, 155*, 27–35. https://doi.org/10.1016/j.ijbiomac.2020.03.174.
559 Li, J., Han, W., Xu, J., Xiong, S., & Zhao, S. (2014). Comparison of morphological changes
560 and *in vitro* starch digestibility of rice cooked by microwave and conductive heating.
561 *Starch/Stärke*, *66*, 549–557. https://doi.org/10.1002/star.201300208.
562 Li, S., Zhang, R., Lei, D., Huang, Y., Cheng, S., Zhu, Z., et al. (2021). Impact of ultrasound,
563 microwaves and high-pressure processing on food components and their interactions.
564 *Trends in Food Science & Technology, 109*, 1–15.
565 https://doi.org/10.1016/j.tifs.2021.01.017.
566 Liazid, A., Guerrero, R. F., Cantos, E., Palma, M., & Barroso, C. G. (2011). Microwave
567 assisted extraction of anthocyanins from grape skins. *Food Chemistry, 124*, 1238–1243.
568 https://doi.org/10.1016/j.foodchem.2010.07.053.

569 Liazid, A., Palma, M., Brigui, J., & Barroso, C. G. (2007). Investigation on phenolic
570 compounds stability during microwave-assisted extraction. *Journal of*
571 *Chromatography A, 1140*, 29–34. https://doi.org/10.1016/j.chroma.2006.11.040.
572 Lu, L. Z., Zhou, Y. Z., Zhang, Y. Q., Ma, Y. L., Zhou, L. X., Li, L., et al. (2010). Anthocyanin
573 extracts from purple sweet potato by means of microwave baking and acidified
574 electrolysed water and their antioxidation *in vitro*. *International Journal of Food*
575 *Science & Technology*, *45*, 1378–1385. https://doi.org/10.1111/j.1365-
576 2621.2010.02271.x.
577 Lu, Y., & Luthria, D. (2016). Influence of gelatinization on the extraction of phenolic acids
578 from wheat fractions. *Food Chemistry*, *194*, 1138–1142.
579 https://doi.org/10.1016/j.foodchem.2015.08.074.
580 Oyeyinka, S. A., Akinware, R. O., Bankole, A. T., Njobeh, P. B., & Kayitesi, E. (2021).
581 Influence of microwave heating and time on functional, pasting and thermal properties
582 of cassava starch. *International Journal of Food Science & Technology, 56*, 215–223.
583 https://doi.org/10.1111/ijfs.14621.
584 Oyeyinka, S. A., Umaru, E., Olatunde, S. J., & Joseph, J. K. (2019). Effect of short microwave
585 heating time on physicochemical and functional properties of Bambara groundnut
586 starch. *Food Bioscience, 28*, 36–41. https://doi.org/10.1016/j.fbio.2019.01.005.
587 Román, L., Martínez, M. M., Rosell, C. M., & Gómez, M. (2015). Effect of microwave
588 treatment on physicochemical properties of maize flour. *Food and Bioprocess*
589 *Technology, 8*, 1330–1335. https://doi.org/10.1007/s11947-015-1493-0.
590 Seoane, P. R, Fernández, N. F, Piñeiro, E. C, & González, H. D (2017). Chapter 6 - Microwave-
591 assisted water extraction. In H. Dominguez González & M. J. González Muñoz (Ed.),
592 *Water Extraction of Bioactive Compounds* (pp. 163–198): Elsevier.
593 https://doi.org/10.1016/B978-0-12-809380-1.00006-1.

Tao, Y., Yan, B., Fan, D., Zhang, N., Ma, S., Wang, L., et al. (2020). Structural changes of starch subjected to microwave heating: A review from the perspective of dielectric properties. *Trends in Food Science & Technology, 99*, 593–607. https://doi.org/10.1016/j.tifs.2020.02.020.

Trancoso-Reyes, N., Ochoa-Martínez, L. A., Bello-Pérez, L. A., Morales-Castro, J., Estévez-Santiago, R., & Olmedilla-Alonso, B. (2016). Effect of pre-treatment on physicochemical and structural properties, and the bioaccessibility of β-carotene in sweet potato flour. *Food Chemistry, 200*, 199–205. https://doi.org/10.1016/j.foodchem.2016.01.047.

Villanueva, M., Harasym, J., Muñoz, J. M., & Ronda, F. (2018). Microwave absorption capacity of rice flour. Impact of the radiation on rice flour microstructure, thermal and viscometric properties. *Journal of Food Engineering, 224*, 156–164. https://doi.org/10.1016/j.jfoodeng.2017.12.030.

Villanueva, M., Harasym, J., Muñoz, J. M., & Ronda, F. (2019). Rice flour physically modified by microwave radiation improves viscoelastic behavior of doughs and its bread-making performance. *Food Hydrocolloids, 90*, 472–481. https://doi.org/10.1016/j.foodhyd.2018.12.048.

Wang, S., Nie, S., & Zhu, F. (2016). Chemical constituents and health effects of sweet potato. *Food Research International, 89*, 90–116. https://doi.org/10.1016/j.foodres.2016.08.032.

Xiang, S., Zou, H., Liu, Y., & Ruan, R. (2020). Effects of microwave heating on the protein structure, digestion properties and Maillard products of gluten. *Journal of Food Science and Technology*, 57, 2139–2149. https://doi.org/10.1007/s13197-020-04249-0.

Xu, Y., Chen, Y., Cao, Y., Xia, W., & Jiang, Q. (2016). Application of simultaneous combination of microwave and steam cooking to improve nutritional quality of cooked

purple sweet potatoes and saving time. *Innovative Food Science & Emerging Technologies, 36*, 303–310. https://doi.org/10.1016/j.ifset.2016.07.014.

Yue, X., & Xu, Z. (2008). Changes of anthocyanins, anthocyanidins, and antioxidant activity in bilberry extract during dry heating. *Journal of Food Science, 73*, C494–C499. https://doi.org/10.1111/j.1750-3841.2008.00845.x.

Zeng, S., Chen, B., Zeng, H., Guo, Z., Lu, X., Zhang, Y., & Zheng, B. (2016). Effect of microwave irradiation on the physicochemical and digestive properties of lotus seed starch. *Journal of Agricultural and Food Chemistry*, *64*, 2442–2449. https://doi.org/10.1021/acs.jafc.5b05809.

Zhang, F., Zhang, Y. Y., Thakur, K., Zhang, J. G., & Wei, Z. J. (2019). Structural and physicochemical characteristics of lycoris starch treated with different physical methods. *Food Chemistry, 275*, 8–14. https://doi.org/10.1016/j.foodchem.2018.09.079.

Zhao, B., Sun, S., Lin, H., Chen, L., Qin, S., Wu, W., et al. (2019). Physicochemical properties and digestion of the lotus seed starch-green tea polyphenol complex under ultrasound-microwave synergistic interaction. *Ultrasonics Sonochemistry*, *52*, 50–61. https://doi.org/10.1016/j.ultsonch.2018.11.001.

Zhu, F. (2015). Interactions between starch and phenolic compound. *Trends in Food Science & Technology, 43*, 129–143. https://doi.org/10.1016/j.tifs.2015.02.003.

Zhu, F., & Sun, J. (2019). Physicochemical and sensory properties of steamed bread fortified with purple sweet potato flour. *Food Bioscience, 30*, 100411. https://doi.org/10.1016/j.fbio.2019.04.012.

Table 1. Chemical composition of purple sweetpotato flour (PSPF) and wheat flour (WF).

Composition	PSPF	WF
Moisture (%)	6.7 ± 0.1	7.2 ± 0.2
Ash (%)	1.7 ± 0.1	0.6 ± 0.1
Protein (%)	5.0 ± 0.4	11.8 ± 0.2
Lipid (%)	0.4 ± 0.0	1.8 ± 0.1
Starch (%)	60.9 ± 0.3	77.1 ± 0.4
Amylose (%)	16.8 ± 0.2	19.8 ± 0.3
Dietary fiber (%)	6.3 ± 0.2	3.3 ± 0.2

Moisture contents were determined by oven drying at 105 °C until constant weight; ash contents were measured using a Muffle furnace at 500 °C; protein contents were measured using a Kjeldahl factor of 6.25; lipid contents were measured by Soxhlet extraction; starch contents were measured using a total starch assay kit (Megazyme, Wicklow, Ireland); amylose contents were measured using an amylose/amylopectin assay kit (Megazyme, Wicklow, Ireland); dietary fiber contents were measured by a total dietary fiber assay kit (Megazyme, Wicklow, Ireland).

Table 2. Particle size and *in vitro* starch digestibility of microwave (MWT) and conventional heat (CHT) treated flours.

Samples	Treatments	Particle size			Starch nutritional fractions		
		D [4, 3] (µm)	D [3, 2] (µm)	d (0.5) (µm)	RDS (%)	SDS (%)	RS (%)
PSPF	MWT 25 °C	183 ± 4c	39.2 ± 0.3d	129 ± 1cd	19.7 ± 0.3d	31.8 ± 0.7b	9.5 ± 0.4b
	MWT 50 °C	172 ± 9d	38.7 ± 1.1d	127 ± 6d	31.4 ± 0.4c	24.6 ± 0.8d	5.0 ± 0.4d
	MWT 100 °C	287 ± 1a	99.7 ± 0.5a	243 ± 1a	35.7 ± 0.3a	15.4 ± 1.0f	9.9 ± 0.6b
	MWT 150 °C	213 ± 1b	75.0 ± 1.3b	174 ± 3b	35.6 ± 0.6a	11.7 ± 0.9g	13.7 ± 0.6a
	CHT 25 °C	176 ± 3cd	34.7 ± 0.6e	102 ± 3f	18.7 ± 0.3de	36.1 ± 0.4 a	6.1 ± 0.6c
	CHT 50 °C	174 ± 2d	38.7 ± 1.2d	124 ± 5d	30.6 ± 0.5c	25.8 ± 0.5c	4.6 ± 0.1d
	CHT 100 °C	183 ± 5c	45.1 ± 0.1c	134 ± 0c	32.6 ± 0.2b	21.7 ± 0.4e	6.6 ± 0.4c
WF	MWT 25 °C	155 ± 11d	35.4 ± 0.7e	119 ± 5d	28.2 ± 0.7d	35.6 ± 0.8b	13.4 ± 0.1d
	MWT 50 °C	183 ± 14c	40.1 ± 0.7d	140 ± 9c	35.3 ± 0.5b	30.0 ± 0.6c	11.8 ± 0.2e
	MWT 100 °C	203 ± 1b	75.5 ± 1.4b	169 ± 5b	40.2 ± 0.5a	20.9 ± 0.6e	16.0 ± 0.2b
	MWT 150 °C	238 ± 6a	85.9 ± 1.8a	174 ± 4b	40.1 ± 0.1a	17.3 ± 0.3f	19.8 ± 0.2a
	CHT 25 °C	118 ± 4e	32.7 ± 0.8f	101 ± 4e	25.1 ± 0.6e	42.2 ± 0.7a	9.8 ± 0.1f
	CHT 50 °C	157 ± 9d	32.9 ± 1.8f	111 ± 7d	34.0 ± 0.9c	29.0 ± 1.2c	14.4 ± 0.3c
	CHT 100 °C	241 ± 1a	71.2 ± 1.1c	191 ± 0a	40.0 ± 0.5a	22.8 ± 0.5d	14.3 ± 0.1c

Values are expressed as mean ± standard deviation of triplicate experiments (n = 3); D [4, 3], volume mean diameter; D [3, 2], surface area mean diameter; d (0.5), number median diameter; RDS, rapidly digestible starch; SDS, slowly digestible starch; RS, resistant starch; values in the same column and of the same flour with the different letters differ significantly ($p < 0.05$).

Table 3. Gelatinization properties and ratio of bands at 1047/1022 cm^{-1} (Fourier-transform infrared spectroscopy (FTIR)) of microwave (MWT) and conventional heat treated (CHT) flours.

Sample	Treatment	T_o (°C)	T_p (°C)	T_c (°C)	ΔH (J/g)	$R_{1047/1022}$
PSPF	MWT 25 °C	54.3 ± 0.7b	63.9 ± 1.6ab	73.5 ± 0.2b	7.84 ± 0.10b	1.031 ± 0.001b
	MWT 50 °C	60.2 ± 0.2a	64.8 ± 0.2a	74.0 ± 0.1ab	7.19 ± 0.15d	1.025 ± 0.003c
	MWT 100 °C	-	-	-	-	1.004 ± 0.001e
	MWT 150 °C	-	-	-	-	1.002 ± 0.002e
	CHT 25 °C	53.3 ± 0.2c	61.7 ± 1.7c	73.5 ± 0.2b	8.10 ± 0.02a	1.046 ± 0.003a
	CHT 50 °C	61.0 ± 0.6a	65.6 ± 0.7a	74.4 ± 0.6a	7.59 ± 0.06c	1.027 ± 0.003c
	CHT 100 °C	-	-	-	-	1.013 ± 0.002d
WF	MWT 25 °C	53.2 ± 0.3c	60.3 ± 0.5c	67.9 ± 0.3b	6.83 ± 0.01b	1.063 ± 0.003b
	MWT 50 °C	59.2 ± 0.2a	62.6 ± 0.1a	68.8 ± 0.4a	5.89 ± 0.09d	1.017 ± 0.001c
	MWT 100 °C	-	-	-	-	1.007 ± 0.002d
	MWT 150 °C	-	-	-	-	1.005 ± 0.001d
	CHT 25 °C	54.5 ± 0.5c	60.5 ± 0.1c	68.8 ± 0.2a	7.37 ± 0.02a	1.075 ± 0.001a
	CHT 50 °C	58.7 ± 0.2b	62.0 ± 0.0b	69.0 ± 0.2a	6.56 ± 0.03c	1.019 ± 0.003c
	CHT 100 °C	-	-	-	-	1.015 ± 0.002c

Values are expressed as the mean ± standard deviation of triplicate experiments (n = 3); T_o, onset temperature; T_p, peak temperature; T_c, conclusion temperature; ΔH, enthalpy change of gelatinization; -, not detectable; values in the same column and of the same flour with the different letters differ significantly ($p < 0.05$).

Table 4. α-Amylase activity, and pasting and gel textural properties of microwave (MWT) and conventional heat (CHT) treated flours.

Samples		α-Amylase activity (CU/g)	Pasting properties					Gel texture properties	
			PV (Pa·s)	HPV (Pa·s)	FV (Pa·s)	BD (Pa·s)	SB (Pa·s)	Hardness (N)	Adhesiveness (N·s)
PSPF	MWT 25 °C	0.80 ± 0.01c	2.35 ± 0.02b	0.18 ± 0.00b	0.27 ± 0.00b	2.17 ± 0.02b	0.10 ± 0.01b	0.041 ± 0.002c	0.201 ± 0.006c
	MWT 50 °C	0.76 ± 0.00d	1.88 ± 0.04d	0.18 ± 0.00b	0.28 ± 0.00b	1.70 ± 0.04d	0.10 ± 0.00b	0.028 ± 0.001d	0.138 ± 0.006d
	MWT 100 °C	0.63 ± 0.03e	-	-	-	-	-	-	-
	MWT 150 °C	0.60 ± 0.00e	-	-	-	-	-	-	-
	CHT 25 °C	0.88 ± 0.01a	3.32 ± 0.01a	0.41 ± 0.00a	0.69 ± 0.00a	2.91 ± 0.02a	0.28 ± 0.01a	0.112 ± 0.002a	0.399 ± 0.005a
	CHT 50 °C	0.83 ± 0.02b	2.08 ± 0.04c	0.14 ± 0.00c	0.22 ± 0.01c	1.94 ± 0.03c	0.08 ± 0.00c	0.060 ± 0.000b	0.280 ± 0.005b
	CHT 100 °C	0.81 ± 0.00bc	-	-	-	-	-	-	-
WF	MWT 25 °C	0.08 ± 0.01b	5.66 ± 0.03b	2.23 ± 0.03b	5.35 ± 0.03b	3.43 ± 0.06a	3.12 ± 0.00b	0.348 ± 0.005b	1.313 ± 0.013b
	MWT 50 °C	0.06 ± 0.00c	5.03 ± 0.03d	2.07 ± 0.01c	4.86 ± 0.06c	2.96 ± 0.01c	2.79 ± 0.07c	0.209 ± 0.001d	0.811 ± 0.014d
	MWT 100 °C	0.06 ± 0.00c	-	-	-	-	-	-	-
	MWT 150 °C	0.04 ± 0.00d	-	-	-	-	-	-	-
	CHT 25 °C	0.09 ± 0.00a	5.79 ± 0.02a	2.34 ± 0.02a	5.54 ± 0.04a	3.45 ± 0.00a	3.20 ± 0.06a	0.375 ± 0.006a	1.452 ± 0.019a
	CHT 50 °C	0.07 ± 0.00b	5.55 ± 0.02c	2.24 ± 0.03b	5.33 ± 0.04b	3.31 ± 0.05b	3.09 ± 0.07b	0.300 ± 0.002c	1.216 ± 0.022c
	CHT 100 °C	0.04 ± 0.01d	-	-	-	-	-	-	-

Values are expressed as the mean ± standard deviation of triplicate experiments (n = 3); CU, Ceralpha unit; PV, peak viscosity; HPV, hot paste viscosity; FV, final viscosity; BD, breakdown viscosity (PV – HPV); SB, setback viscosity (FV – HPV); -, not detectable; values in the same column and of the same flour with the different letters differ significantly ($p < 0.05$).

Table 5. Color, total phenolic content (TPC) and *in vitro* antioxidant potential of microwave (MWT) and conventional heat (CHT) treated flours.

Samples	Treatments	Color					TPC	FRAP
		*L**	*a**	*b**	Δ*C*	Δ*E*	(g GAE/kg db)	(mmol TE/kg db)
PSPF	MWT 25 °C	63.0 ± 0.2ab	16.9 ± 0.2b	–2.20 ± 0.13b	17.0 ± 0.1d	65.3 ± 0.2a	6.43 ± 0.14e	58.2 ± 1.0c
	MWT 50 °C	62.7 ± 0.8ab	18.4 ± 0.3a	–2.58 ± 0.13c	18.6 ± 0.3b	65.4 ± 0.7a	8.17 ± 0.17d	70.2 ± 2.2b
	MWT 100 °C	56.7 ± 0.2d	18.7 ± 0.3a	–5.44 ± 0.02e	19.5 ± 0.3a	60.0 ± 0.2d	11.04 ± 0.16b	77.3 ± 0.4a
	MWT 150 °C	62.2 ± 0.5b	11.5 ± 0.1e	7.37 ± 0.22a	13.6 ± 0.2f	63.7 ± 0.4b	12.31 ± 0.16a	77.8 ± 0.3a
	CHT 25 °C	63.4 ± 0.6a	14.7 ± 0.1d	–6.22 ± 0.09g	15.9 ± 0.0e	65.4 ± 0.6a	4.20 ± 0.10g	34.1 ± 2.4e
	CHT 50 °C	63.7 ± 0.3a	15.1 ± 0.1c	–4.83 ± 0.07d	15.9 ± 0.1e	65.6 ± 0.3a	5.89 ± 0.20f	41.7 ± 2.1d
	CHT 100 °C	60.1 ± 0.8c	16.6 ± 0.1b	–5.93 ± 0.05f	17.6 ± 0.1c	62.6 ± 0.8c	9.73 ± 0.02c	75.3 ± 0.7a
WF	MWT 25 °C	93.6 ± 0.3b	5.00 ± 0.03ab	4.00 ± 0.05d	6.40 ± 0.03d	93.8 ± 0.3b	-	-
	MWT 50 °C	92.2 ± 0.3d	5.05 ± 0.03a	4.78 ± 0.09ab	6.95 ± 0.05a	92.4 ± 0.3d	-	-
	MWT 100 °C	94.9 ± 0.1a	4.55 ± 0.01e	4.90 ± 0.05a	6.69 ± 0.02c	95.1 ± 0.1a	-	-
	MWT 150 °C	90.6 ± 0.1e	4.78 ± 0.04d	4.72 ± 0.05b	6.72 ± 0.04bc	90.8 ± 0.1e	-	-
	CHT 25 °C	93.8 ± 0.1b	4.94 ± 0.04bc	4.12 ± 0.04d	6.43 ± 0.05d	94.0 ± 0.1b	-	-
	CHT 50 °C	92.8 ± 0.3c	4.90 ± 0.04c	4.69 ± 0.11b	6.79 ± 0.07b	93.0 ± 0.3c	-	-
	CHT 100 °C	92.7 ± 0.3c	4.20 ± 0.10f	4.35 ± 0.12c	6.04 ± 0.02e	92.9 ± 0.3cd	-	-

Values are expressed as the mean ± standard deviation of triplicate experiments (n = 3); *L**, lightness; *a**, redness-greenness; *b**, yellowness-blueness; Δ*C*, chroma value; Δ*E*, color intensity; FRAP, ferric reducing ability of plasma; GAE, gallic acid equivalents; TE, Trolox equivalents; db, dry basis; -, not detectable; values in the same column and of the same flour with the different letters differ significantly ($p < 0.05$).

Table 6. Composition (g CAE/kg db) of hydroxycinnamic acid derivatives (HAD) in microwave (MWT) and conventional heat (CHT) treated PSPF.

		Concentration (g CAE/kg db)										
Sample	Treatment	3-CQA	5-CQA	4-CQA	CA	FQA	4,5-diCQA	3,5-diCQA	3,4-diCQA	CFQA	diCFQA	Total HAD
PSPF	MWT 25 °C	0.19 ± 0.00e	1.18 ± 0.02d	0.05 ± 0.00e	0.16 ± 0.00e	0.15 ± 0.00c	0.26 ± 0.01e	1.75 ± 0.01c	0.29 ± 0.01e	0.11 ± 0.00b	0.17 ± 0.00e	4.30 ± 0.06e
	MWT 50 °C	0.22 ± 0.00d	1.22 ± 0.02c	0.06 ± 0.00d	0.18 ± 0.00e	0.15 ± 0.00c	0.39 ± 0.01d	1.95 ± 0.02b	0.43 ± 0.01d	0.12 ± 0.00a	0.21 ± 0.00d	4.93 ± 0.07d
	MWT 100 °C	0.37 ± 0.00b	1.63 ± 0.02a	0.47 ± 0.00b	0.30 ± 0.03a	0.15 ± 0.00c	1.93 ± 0.02a	2.04 ± 0.02a	2.07 ± 0.03a	0.09 ± 0.00d	0.50 ± 0.01a	9.55 ± 0.07a
	MWT 150 °C	0.74 ± 0.00a	0.78 ± 0.00f	0.74 ± 0.01a	0.26 ± 0.00bc	0.04 ± 0.00e	1.39 ± 0.01b	0.94 ± 0.01f	1.40 ± 0.01c	0.04 ± 0.00f	0.28 ± 0.00c	6.61 ± 0.05c
	CHT 25 °C	0.16 ± 0.00f	0.74 ± 0.02g	0.02 ± 0.00f	0.25 ± 0.00c	0.19 ± 0.00a	0.12 ± 0.01g	1.57 ± 0.03d	0.14 ± 0.01g	0.10 ± 0.00c	0.11 ± 0.00g	3.38 ± 0.07g
	CHT 50 °C	0.22 ± 0.00d	0.92 ± 0.02e	0.06 ± 0.00d	0.28 ± 0.00ab	0.17 ± 0.00b	0.17 ± 0.01f	1.47 ± 0.02e	0.20 ± 0.01f	0.09 ± 0.00d	0.12 ± 0.00f	3.70 ± 0.06f
	CHT 100 °C	0.32 ± 0.00c	1.27 ± 0.02b	0.39 ± 0.01c	0.22 ± 0.00d	0.14 ± 0.00d	1.33 ± 0.02c	1.50 ± 0.02e	1.50 ± 0.03b	0.07 ± 0.00e	0.32 ± 0.01b	7.04 ± 0.10b

Values are expressed as the mean ± standard deviation of triplicate experiments (n = 3); 3-CQA, 3-caffeoylquinic acid; 5-CQA, 5-caffeoylquinic acid; 4-CQA, 4-caffeoylquinic acid; CA, caffeic acid; FQA, feruloylquinic acid; 4,5-diCQA, 4,5-dicaffeoylquinic acid; 3,5-diCQA, 3,5-dicaffeoylquinic acid; 3,4-diCQA, 3,4-dicaffeoylquinic acid; CFQA, caffeoyl-feruloylquinic acid; diCFQA, dicaffeoyl-feruloylquinic acid; CAE, chlorogenic acid equivalents; db, dry basis; values in the same column with different letters differ significantly ($p < 0.05$).

Table 7. Composition (mg CGE/kg db) of anthocyanins (AC) in microwave (MWT) and conventional heat (CHT) treated PSPF.

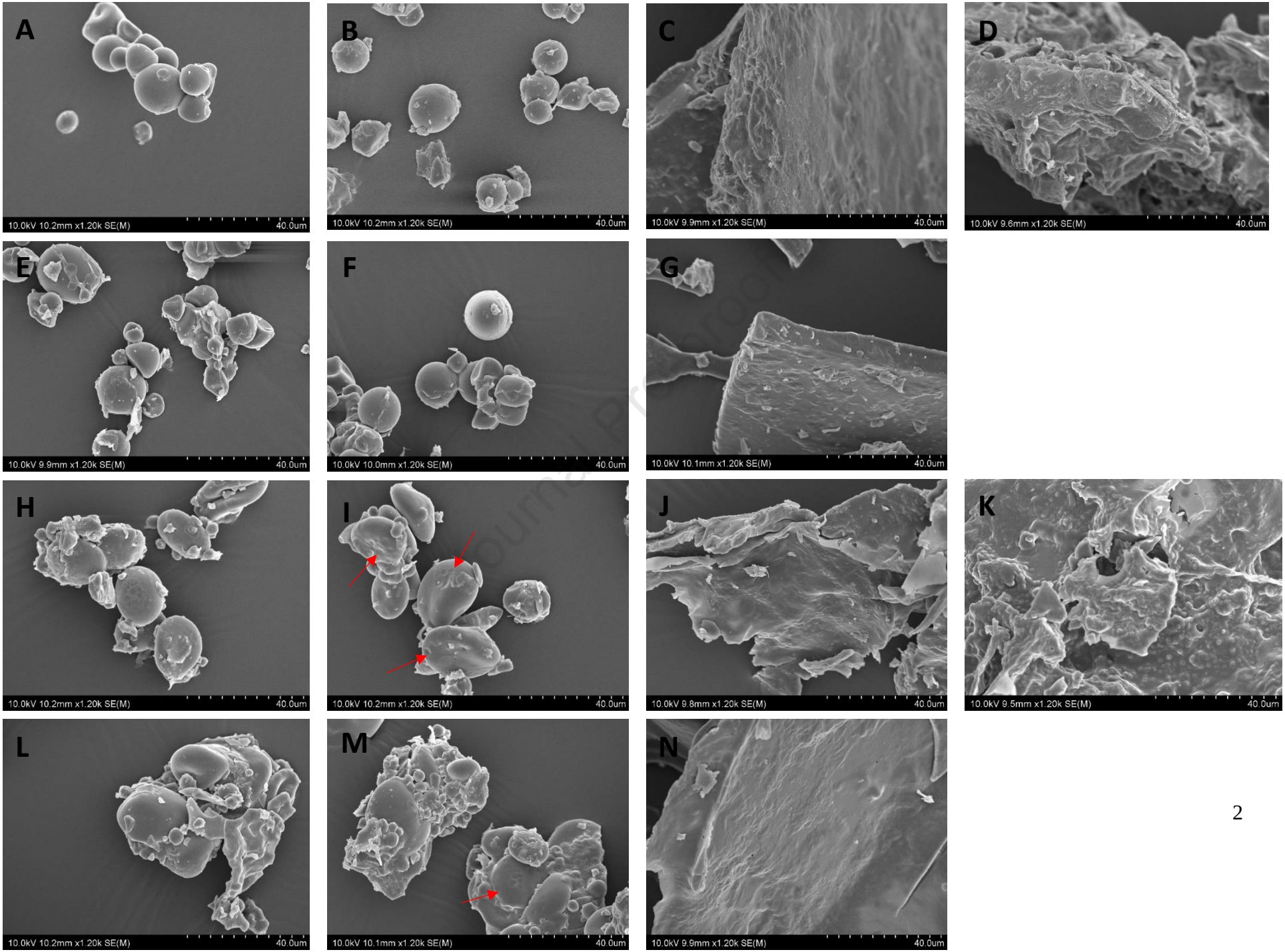
Sample	Treatment	Concentration (mg CGE/kg db)							
		cy 3-*p*-hydroxybenzoylsoph-5-glc	pn 3-*p*-hydroxybenzoylsoph-5-glc	cy 3-(6"-feruloylsoph)-5-glc	pn 3-(6"-feruloylsoph)-5-glc	cy 3-(6"-caffeoyl-6"'-feruloylsoph)-5-glc	pn 3-(6"-caffeoyl-6"'-*p*-hydroxybenzoylsoph)-5-glc	pn 3-(6"-caffeoyl-6"'-feruloylsoph)-5-glc	Total AC
PSPF	MWT 25 °C	4.0 ± 0.3d	28.7 ± 0.8d	135 ± 2e	4.5 ± 0.4c	-	180 ± 0c	6.0 ± 0.0a	358 ± 3d
	MWT 50 °C	7.8 ± 0.9c	58.0 ± 0.6b	159 ± 3c	14.1 ± 1.0a	5.0 ± 2.0d	172 ± 1c	-	415 ± 2c
	MWT 100 °C	15.2 ± 0.7a	12.2 ± 0.2e	232 ± 7a	10.7 ± 2.3b	60.0 ± 3.0a	682 ± 2a	5.0 ± 0.0b	1017 ± 48a
	MWT 150 °C	-	-	35 ± 1g	4.7 ± 0.0c	6.0 ± 0.0cd	88 ± 0d	6.0 ± 0.0a	140 ± 1f
	CHT 25 °C	-	2.6 ± 0.8f	109 ± 2f	-	-	154 ± 0c	-	265 ± 2e
	CHT 50 °C	-	45.0 ± 0.6c	146 ± 3d	-	-	158 ± 0c	-	350 ± 14d
	CHT 100 °C	8.2 ± 0.2b	63.0 ± 0.4a	216 ± 7b	9.4 ± 0.7b	9.0 ± 0.0b	294 ± 1b	5.0 ± 0.0c	606 ± 17b

Values are expressed as the mean ± standard deviation of triplicate experiments (n = 3); cy, cyanidin; soph, sophoroside; glc, glucoside; pn, peonidin; CGE, cyanidin-3-glucoside equivalents; db, dry basis; -, not detected; values in the same column with different letters differ significantly ($p < 0.05$).

Figure caption

Figure 1. Scanning electron microscopy (SEM) images of purple sweetpotato flour (PSPF) and wheat flour (WF) affected by microwave treatment (MWT) and conventional heat treatment (CHT). (A), PSPF treated by MWT at 25 °C; (B), PSPF treated by MWT at 50 °C; (C), PSPF treated by MWT at 100 °C; (D), PSPF treated by MWT at 150 °C; (E), PSPF treated by CHT at 25 °C; (F), PSPF treated by CHT at 50 °C; (G), PSPF treated by CHT at 100 °C; (H), WF treated by MWT at 25 °C; (I), WF treated by MWT at 50 °C; (J), WF treated by MWT at 100 °C; (K), WF treated by MWT at 150 °C; (L), WF treated by CHT at 25 °C; (M), WF treated by CHT at 50 °C; (N), WF treated by CHT at 100 °C.

Notes: The accelerating voltage was 10.0 kV; the working distance was around 10.0 mm; the magnification was 1.20 k; the scale bar was 40 μm; red arrows indicated the MWT or CHT effects on starch granules.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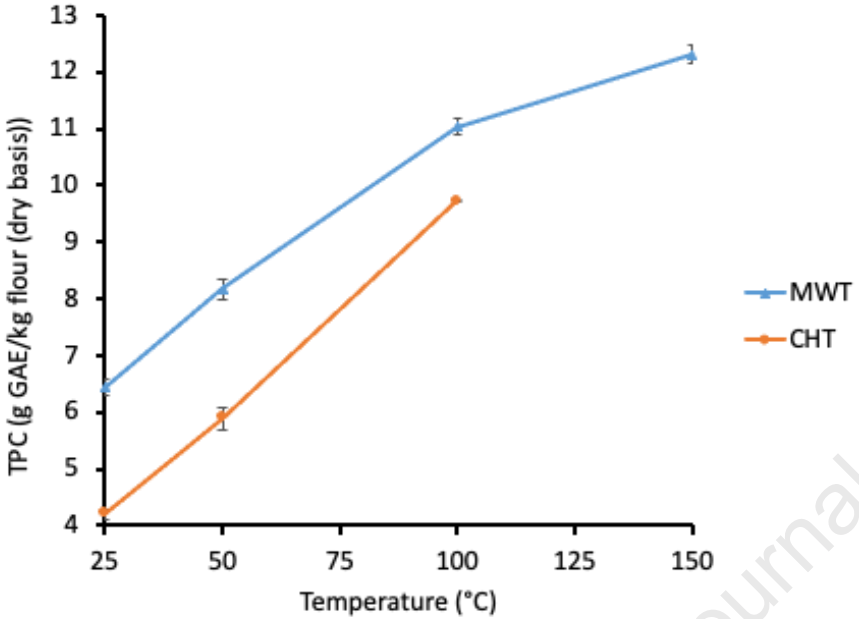
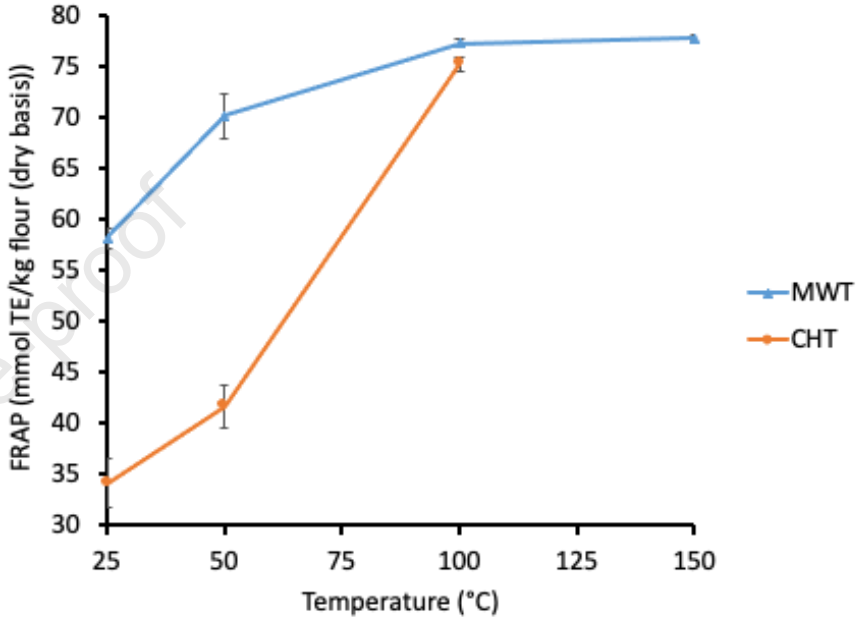
B
10.0kV 10.2mm x1.20k SE(M) 40.0um
C
10.0kV 9.9mm x1.20k SE(M) 40.0um
D
10.0kV 9.6mm x1.20k SE(M) 40.0um
E
10.0kV 9.9mm x1.20k SE(M) 40.0um
F
10.0kV 10.0mm x1.20k SE(M) 40.0um
G
10.0kV 10.1mm x1.20k SE(M) 40.0um
H
10.0kV 10.2mm x1.20k SE(M) 40.0um
I
10.0kV 10.2mm x1.20k SE(M) 40.0um
J
10.0kV 9.8mm x1.20k SE(M) 40.0um
K
10.0kV 9.5mm x1.20k SE(M) 40.0um
L
10.0kV 10.2mm x1.20k SE(M) 40.0um
M
10.0kV 10.1mm x1.20k SE(M) 40.0um
N
10.0kV 9.9mm x1.20k SE(M) 40.0um

Figure 2. Total phenolic content (TPC) and *in vitro* antioxidant activity (FRAP) of purple sweetpotato flour affected by microwave treatment (MWT) and conventional heat treatment (CHT). GAE, gallic acid equivalent; TE, Trolox equivalent.





- Microwave treatment (MWT) exerted different effects according to the temperature
- MWT gave more damages on starch granules that conventional heat treatment (CHT)
- MWT had more pronounced effect on flour viscosity than conventional heat treatment
- The digestibility of microwaved flour was higher than conventional heated flour
- MWT gave higher extraction efficiency of anthocyanins (by 68%) than CHT at 100 °C

Declaration of interests

☒The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: