



Retaining a large amount of resistant starch in cooked potato through microwave heating after freeze-drying

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ABSTRACT

Resistant starch (RS) is beneficial for humans, especially for the diabetics. Raw potato had a great deal of RS, while most of which become digestible after gelatinization. Thus, few RS will be retained in potatoes after regular cooking. To preserve RS in cooked potatoes as much as possible, microwave heating before (MFD) and after freeze-drying (FDM) were conducted with three different potatoes. After MFD, the RS content in potatoes was lower than 7% and the RDS content was higher than 45% for three potatoes. However, RS in potatoes treated with FDM was still as high as 40%, similar to that in the raw potatoes. Meantime, FDM caused less browning, produced a certain level of pyrazines, benzeneacetaldehyde and other flavor compounds, endowing cooked potatoes special baked flavor. Freeze-drying before microwave heating is a valuable way to reserve RS in cooked potatoes, which could also be used to reserve high RS content in crisp, chips, and other processed potatoes.

1. Introduction

Changes of diets and lifestyle in modern society cause increasing prevalence of type 2 diabetes (T2D) (Krug, 2016), of which the population is estimated to reach up to 439 million by 2030 (Liu et al., 2020). Diet high in sugar may raise insulin level, cause obesity and T2D (Cust et al., 2009). Intaking of starch is reported to be related to the occurrence of T2D, while diet rich in dietary fiber is associated with a lower risk of T2D, the quality of carbohydrate is believed to be associated with the risk of T2D (AlEsa et al., 2015).

Starch, the main dietary carbohydrate, can be classified into 3 types according to its digestibility: rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS) (Aller et al., 2011). RDS is rapidly digested and absorbed in the small intestine after ingestion, leading to a quick elevation of blood glucose. SDS is slowly digested and can maintain a slow release of glucose, leading to a prolonged energy availability. RS, known as a novel kind of dietary fiber, cannot be digested in the intestine of a healthy person and is fermented by gut microbiota in colon to produce short-chain fatty acids that are beneficial for human (Aller et al., 2011). According to the mechanism of its enzymatic resistance, RS is classified into RS1, RS2, RS3, RS4 and RS5. Among which RS2 refers to the native or ungelatinized granules such as

raw potato or banana starches, and RS3 presents the retrograded starch (Liu et al., 2020). Studies showed that RS can control the postprandial glycemic response, increase insulin sensitivity and thus help to prevent T2D and obesity (Liu et al., 2020), but the content of RS varied considerably among starches derived from different botanical origins.

Potato (*Solanum tuberosum* L.) is rich in starch, dietary fiber, biologically active compounds and is the most commonly consumed non-cereal staple food worldwide. Approximately half of potatoes produced in the world are consumed fresh, and the remaining are majorly used in food industry as functional food ingredient and animal feeding (Birch et al., 2012). Raw potato contains about 47–59% RS of its dry matter, and is a natural source of RS (Robertson et al., 2018). However, the majority of RS in raw potatoes is RS2, which usually presents as a B- or C-type polymorph (Liu et al., 2020) and becomes highly digestible after gelatinization (Robertson et al., 2018). Thus, cooked potatoes or potato products prepared with common cooking processes contain very low RS (2–4% of dry matter) (Robertson et al., 2018), abundant RS in raw potatoes is hardly utilized.

To maintain RS in processed potatoes as much as possible, many improved processing methods have been attempted, such as cooking with different heating sources, moisture content, cooking time, and heating intensity (Evans and Haisman, 1982; Narwojsz et al., 2020; Tao

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et al., 2020). Compared to boiling, steaming and other heating methods, microwave heating was better in keeping RS in potatoes (Narwojsz et al., 2020; Tao et al., 2020). Under heating, starch granules undergo gelatinization at the presence of water, but the extent of gelatinization is limited by the moisture content (Evans and Haisman, 1982). Lower water content may restrain the gelatinization of starch and increase the enzymatic resistance, freeze-drying under vacuum is a suitable method to lower the water content in potatoes with less browning (Narwojsz et al., 2020). Microwave heating combined with freeze-drying was supposed to be a potential way to keep major RS in cooked potato. In this study, the physiochemical properties and the flavors in potatoes with microwave heating before or after freeze-drying were investigated, RS content in potatoes microwave heated after freeze-drying was as high as that in raw potatoes. Freeze-drying before microwave heating had been found to be an effective process to maintain rich RS in cooked potatoes.

2. Materials and methods

2.1. Materials

Three kinds of fresh potatoes (crumby, waxy, floury) were purchased from a supermarket (Albert Heijn, Wageningen, Netherlands). After washing and peeling, the potatoes were quickly cut into cubes with 1 cm³ then microwave heating before or after freeze-dried; Freeze-drying was conducted below −55 °C and 10 Pa for 48 h with a freeze-drier (FreeZone, Labconco, USA). The heating of the microwave (MD10495, Medion AG, Germany) was set at 700 W for 4 min, with which the potatoes were well cooked but not burnt. Potatoes freeze-dried only were used as control. Part of potato samples was milled using ball miller and sieved through 80 mesh sieves. All samples were kept in sealed package at 4 °C till use. For simplification, FD, MDF, FDM in the following sections represent treatments of freeze-drying only, microwave heating followed by freeze-drying and microwave heating after freeze-drying respectively.

2.2. Color measurement

The color of potatoes with different treatments was measured according to the methods described by Ashwar et al. (2016) and Wang et al. (2020) using a Color Flex spectrophotometer (Hunter Associates Laboratory, Inc., Reston, USA). A green tile was used to standardize the color meter. After that, the L (lightness), a (redness), b (yellowness) values and total color difference (ΔE) of the samples were measured. The total color difference (ΔE) was calculated according to the method described by Ashwar et al. (2016). Each sample was measured five times.

2.3. Measurement of starch fractions

Total starch was measured with total starch kit (K-TSTA) according to its' manual procedure respectively. RS, RDS and SDS content in potato flours were measured according to the methods described by Englyst et al. (2006), and the result was presented as the percentage of dry matter of the potato flour.

2.4. In vitro digestion

The *in vitro* digestion properties of the samples were measured using a standardized INFOGEST experimental protocol as described by Minekus et al. (2014) with minor modifications. The oral phase step was omitted. Briefly, 0.5 g of potato flour was weighed into 50 mL tubes, then 5 mL simulated salivary fluid, 25 μ L CaCl₂ (0.3M) and 1.48 mL MQ water were added and mixed thoroughly.

For the gastric phase, 7.5 mL simulated gastric fluids, 1.6 mL pepsin (25000 U/mL), and 5 μ L CaCl₂ (0.3M) were added to the tubes. After that, the pH of the samples was adjusted to 3 with 1M HCl. Then the

samples were rotated at 20 rpm for 2 h at 37 °C.

For the intestinal phase, the samples were mixed with 11 mL simulated intestinal fluids, 2.5 mL bile salt solution (160 mM), and 40 μ L CaCl₂ (0.3M). Then pH of the samples was adjusted to 7 with 1M NaOH. After that, pancreatin (the α -amylase activity in the final volume of 40 mL intestinal mixture was controlled at 200 U/mL) of 5 mL were added, the mixture was rotated at 20 rpm at 37 °C for 2 h. During the stimulated intestinal phase, sample of 100 μ L was taken out to 1.5 mL tubes at 10-time points (0, 10, 20, 30, 45, 60, 90, 120, 150, and 180 min). Then mixed with 0.4 mL of ethanol and vortexed immediately to inactivate the enzyme. The ethanolic mixtures were then centrifuged (4000 \times g, 15 min) and the digested starch in the supernatant were determined according to the procedures described as Shen et al. (2021), the digestion data was then fitted to a first-order model (Goñi et al., 1997) as below:

$$C_t = C_\infty (1 - e^{-kt})$$

Where C_t , C_∞ is the concentration of the percentage of digested starch at a certain time (t) and infinite time, k is the digestibility rate constant.

The estimated glycemic index (GI) was measured by comparing with white bread as a reference food (Goñi et al., 1997). The reference sample was digested following the same procedure as the test samples. The estimated glycemic index (eGI) was calculated by following equation:

$$eGI = 39.71 + 0.549 \times \frac{iAUC90_{test}}{iAUC90_{ref}} \times 100$$

Where $iAUC90_{test}$, $iAUC90_{ref}$ is the area under the digestion curve up to 90 min of the test sample, the reference sample, respectively.

2.5. Texture properties

Texture properties of potato cubes were measured using a TA-XT texture analyzer (Stable Micro System, Surrey, UK) with a 5 mm diameter cylinder probe. The pre-speed, post-speed, and test speed were set as 2 mm/s. The penetration distance was set as 10 mm and the trigger force was 20 g. Texture properties were derived from the force–deformation curve (force versus time). The maximum force of break and the first significant force of break were indicated as hardness and fracturability respectively. For each treatment, measurements were taken on five different cubes individually and the average values were reported.

2.6. Differential scanning calorimetry (DSC)

Thermal properties of potato flours were measured with a differential scanning calorimeter (TA Q200, New Castle, USA) according to Sun et al. (2018). Briefly, sample of 2.5 mg was weighed into an aluminum DSC pan, then 7.5 μ L of deionized water was added. The pan was hermetically sealed and equilibrated for 12 h at room temperature. The samples were then scanned at a heating rate of 4 °C/min from 30 to 130 °C with a hermetically sealed empty pan as a reference. The onset temperature (T_o), peak gelatinization temperature (T_p), and enthalpy changes (ΔH) were determined with Universal Analysis 2000 program (Version 4.4A) software supplied with the instrument.

2.7. X-ray diffraction (XRD)

The crystallinity was determined with an X-ray diffractometer (D8 Advance, Bruker, Germany) which was equipped with a copper tube operating at 40 kV and 40 mA and producing Cu K α radiation ($\lambda = 0.154$ nm). Diffractograms were obtained by scanning from 5° to 40° (2θ) with a step of 0.2°. The relative crystallinity was calculated with DIFFRAC.EVA V5.2 software.

2.8. GC-MS

Flavor compounds of the samples were measured using GC-MS

(PerkinElmer Clarus 500, Norwalk, USA) according to the method described by Ekpa et al. (2020). Briefly, potato flour of 1.5 g was weighed into a 10 mL glass vial and incubated at 40 °C for 15 min. The SPME fiber was desorbed in the GC injector port at 250 °C for 10 min. The extracted volatile compounds were sent into the Stabilwax DA capillary column (30 m × 0.25 mm, ID × 0.25 μm) by helium as carrier gas at 1 mL/min. The separation program for SPME injection was 40 °C for 2 min, increased at 10 °C/min to 230 °C, and then held at 230 °C for 5 min. In the end, the separated compounds were eluted through 230 °C mass-transfer lines, and the mass spectrum of the individual compound was detected by the mass detector operating in an electron ionization mode (internal ionization source; 70 eV) with a scan range from 33 to 300 m/z.

Mass spectrums of each GC peak were searched in the NIST (National Institute of Standards and Technology) database. Flavor compounds were identified and their peak areas were automatically calculated by Xcalibur 4.0 (Thermo Fisher Scientific Inc.). In order to filter out noise peaks, only peaks with area ranked in the top 40 were considered. Best hit compound(s) of a certain GC peak with SI ≥ 900 or RSI ≥ 900 while Prob or combined Prob ≥ 60% were selected. The relative abundance of the compounds was calculated as average peak area.

2.9. Statistical analysis

All the experiments were triplicate except INFOGEST *in vitro* digestion was duplicated. The data were subjected to one-way analysis of variance (ANOVA) followed by post-hoc Turkey multiple range tests with SAS (v9.1, SAS Institute Inc., NC, USA), and SPSS (v26.0, SPSS Inc., Chicago, USA). A principal components analysis (PCA) of all parameters determined in this study (except flavor components) was conducted by R (v4.2.0) script.

3. Results and discussions

3.1. Color of processed potatoes

Color of processed potatoes is an important index that is tightly related to consumer preferences (Pedreschi et al., 2007). All three potatoes after FD had similar L, a, and b values (Table 1). Potatoes treated with MFD became darker and had a lower L, a, but higher b, while FDM caused higher a and b values but unchanged L, except for crumbly potatoes which had lower L when compared to FD. Generally, the changes

Table 1
Color parameters of three potatoes with different treatment^a.

Materials	Treatment	L	a	b	Δ E
crumbly potato	FD	87.8 ± 0.1 ^a	-0.38 ± 0.08 ^b	12.1 ± 0.1 ^a	
	MFD	84.5 ± 1.0 ^b	-0.83 ± 0.31 ^c	22.0 ± 0.3 ^c	10.5 ± 0.2
	FDM	82.3 ± 0.8 ^c	3.32 ± 0.30 ^a	20.7 ± 0.4 ^b	10.9 ± 0.8
waxy potato	FD	88.1 ± 0.1 ^a	-0.53 ± 0.02 ^c	18.0 ± 0.2 ^a	
	MFD	84.6 ± 0.4 ^b	-0.26 ± 0.18 ^b	26.9 ± 0.6 ^c	9.6 ± 0.6
	FDM	88.1 ± 0.1 ^a	0.04 ± 0.01 ^a	19.4 ± 0.1 ^b	1.5 ± 0.1
floury potato	FD	88.0 ± 0.0 ^a	0.09 ± 0.04 ^b	14.6 ± 0.1 ^a	
	MFD	83.8 ± 0.3 ^b	-0.65 ± 0.08 ^c	22.1 ± 0.2 ^c	8.6 ± 0.2
	FDM	88.0 ± 0.2 ^a	0.20 ± 0.03 ^a	15.2 ± 0.1 ^b	0.7 ± 0.0

^a The same small letters indicated there were no significant differences among different treatments for the same variety. FD: Freeze-drying, MFD: microwave heating before freeze-drying treatment, FDM: microwave heating before freeze-drying treatment.

of colors in FDM potatoes were less as shown by a smaller ΔE, except for crumbly potato. The lower lightness in MFD potatoes might be due to the starch gelatinization and uptake of water during heating, which was also observed in blanched potato samples (Wang et al., 2010). When there was lower moisture content, microwave treatment caused subtle changes in the color of the starch, as also observed by Kumar et al. (2020). Crumbly potato was more sensitive to heating, its browning caused by microwave treatment was more serious than the other two potatoes, Color 'a' seems to be the major contributor for the difference between crumbly FDM potatoes and other FD and FDM potatoes (Fig. 1). The browning of the potatoes may be caused by the Maillard reaction (Pedreschi et al., 2007).

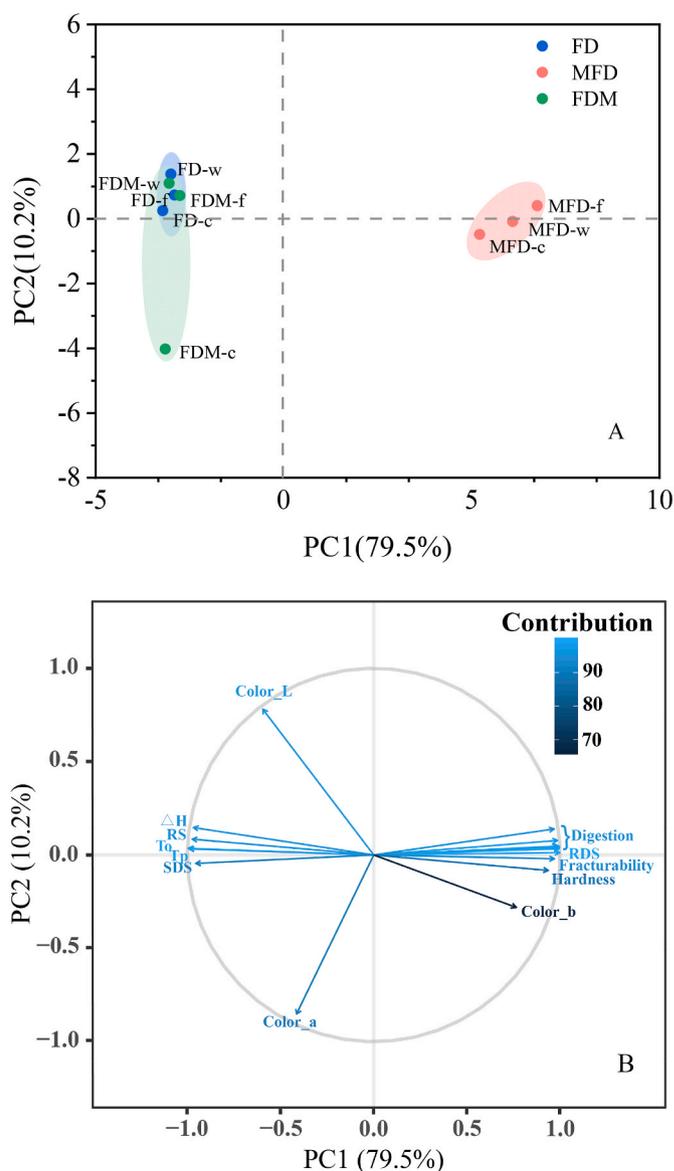


Fig. 1. Principal component analysis of all obtained data. (A) Component scores to show the correlations among different samples. (B) Component loadings to show physicochemical parameters correlation. Only the first two main factors were released; PC1 explained the majority variances among all variables. Arrows indicate main trends in correlations between the different measured variables in the three treatments. Variables in the same directions showed positive relationship, in opposite showed negative relationship and in orthogonal directions varied independently.

3.2. Total starch, RS content

Total starch content of three potatoes all was around 60% (Table 2), which was in line with that reported by Robertson et al. (2018). The RS content in crumbly, waxy, and flourey potato was 40%, 36%, 38% and the RDS content was 8%, 10%, 9%, respectively. After MFD, the RS content in three potatoes were decreased dramatically (lower than 12%) and the RDS content were increased greatly (higher than 45%) compared to FD. While the RS and RDS content in potatoes treated with FDM were similar to that in FD potatoes (Table 2). RS and RDS are decisive for differentiating potatoes with different treatments, MFD potatoes had lower RS and higher RDS content were clustered alone along PC1 which explain 79.5% variations between FD, FDM and MFD potatoes (Fig. 1). The great decreases in RS of fresh potato caused by microwave treatment were also observed by other studies (Yang et al., 2016; Narwojsz et al., 2020). During heating at the presence of certain moisture, hydrogen bonds between amylose chains and amylopectin branched chains are cleaved, and hydrogen bonds between starch and water are strengthened, leading to a changed starch crystalline structure (Narwojsz et al., 2020) and increased susceptible to enzymatic hydrolysis. On the other hand, few waters remained after freeze-drying and the granule crystalline was affected subtly by subsequent microwave heating, thus the RS content in potatoes treated with FDM did not change significantly.

3.3. Starch in vitro digestion

In vitro digestion showed that the MFD potatoes had larger C_{∞} and k than that of potatoes treated with FD and FDM, exhibited higher digestion rate and extent, while FDM potatoes showed similar digestibility as FD potatoes (Fig. 2). The In vitro digestion rate showed negative correlation to the content of RS and SDS, and contributed greatly to the PC1 (Fig. 1). Correspondingly, the eGI of three potatoes were all below 51 (47.2, 50.9, 50.8 for crumbly, waxy, and flourey potatoes respectively), potatoes treated with FDM had similar low eGI as the raw potatoes. While after treating with MFD, the eGI of crumbly, waxy, and flourey potato increased pronouncedly to 83.1, 89.8, and 93.6 respectively (Fig. 2). Compared to high GI (>70) foods, foods with low (<55) and medium GI (56–69) are slowly digested and absorbed by the gut and could maintain a stable postprandial blood glucose level (Thiranosornkij et al., 2019). These results suggested that freeze-drying

Table 2
RS, SDS, and RDS of three potatoes with different treatment.

Variety	Treatment	RDS (%)	SDS (%)	RS (%)	Total starch (%)
crumbly potato	FD	8.3 ± 0.5 ^a	11.9 ± 1.4 ^a	40.3 ± 1.4 ^a	60.5 ± 1.3 ^a
	MFD	45.2 ± 3.2 ^b	5.6 ± 0.4 ^b	11.1 ± 3.6 ^b	61.8 ± 0.4 ^a
	FDM	8.0 ± 1.8 ^a	14.0 ± 1.9 ^a	33.7 ± 1.9 ^c	55.6 ± 1.0 ^b
waxy potato	FD	9.8 ± 0.9 ^a	13.8 ± 1.6 ^a	36.3 ± 1.6 ^a	59.9 ± 0.4 ^a
	MFD	46.5 ± 5.4 ^b	6.6 ± 3.1 ^b	6.2 ± 4.1 ^b	59.2 ± 3.4 ^a
	FDM	11.1 ± 0.6 ^a	14.8 ± 0.9 ^a	32.3 ± 0.9 ^a	58.1 ± 1.7 ^a
flourey potato	FD	9.4 ± 0.6 ^a	11.6 ± 0.8 ^a	38.1 ± 0.8 ^a	59.1 ± 0.6 ^a
	MFD	50.3 ± 1.4 ^b	4.9 ± 2.5 ^b	8.8 ± 1.2 ^b	64.1 ± 0.8 ^{ab}
	FDM	8.1 ± 0.7 ^a	11.9 ± 0.8 ^a	41.5 ± 0.8 ^c	61.5 ± 0.6 ^b

*: The same small letters indicated there were no significant differences among different treatments for the same variety. FD: Freeze-drying, MFD: microwave heating before freeze-drying treatment, FDM: microwave heating before freeze-drying treatment.

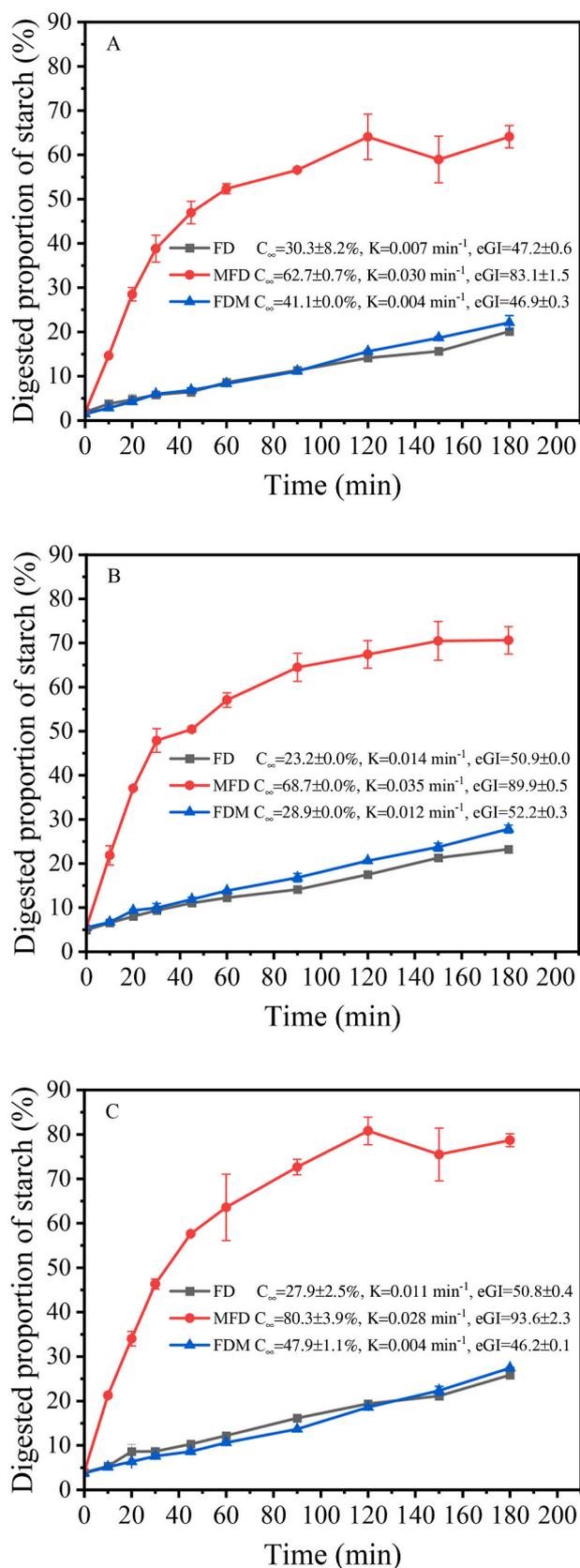


Fig. 2. In vitro starch digestion of three potatoes with different treatments. A: Crumbly potato; B: waxy potato; C: flourey potato. FD: Freeze-drying, MFD: microwave heating before freeze-drying treatment, FDM: microwave heating before freeze-drying treatment.

before heating can maintain a high RS content in cooked potatoes, and freeze-dried potatoes can be processed into low-GI products.

3.4. Texture properties

Freeze-dried waxy potato and flourey potato had similar fracturability (104 and 91 g respectively), while crumbly potato had a slightly higher fracturability of 137 g. All three potatoes had similar hardness, which were 209, 226, 218 g for crumbly, waxy, and flourey potatoes respectively. After MFD, the fracturability of crumbly, waxy, and flourey potatoes was 357, 344, and 308 g respectively, and the hardness was 619, 555, and 466 respectively, significantly higher than those of FD samples (Table 3), indicating MFD resulted in a harder and more fragile texture. While FDM caused no significant changes in fracturability and hardness for all three potatoes. The fracturability and hardness of potatoes showed positive correlation with the digestion rate and negative correlation to the RS and SDS (Fig. 1). The increases in fracturability and hardness caused by MFD might be because of the gelatinization of starch in the presence of free water upon heating, besides the deformation and disintegration of the cell wall, the starch granule structure became dense due to water sublimation during heating (Bondaruk et al., 2007; Wang et al., 2010). While FD removes the majority of the free water, thus the gelatinization of starch granules during microwave heating was limited and FD potatoes maintained similar texture as the raw potatoes (Table 3).

3.5. Thermal properties

For three potatoes after freeze-drying, T_o were spanned from 59.6 to 60.3 °C, T_p were ranged from 62.9 to 64.3 °C, and ΔH were ranged from 12.0 to 13.0 J/g, there were no significant differences in T_o , T_p , and ΔH among three potatoes. FDM result in slight changes in T_o , T_p , and ΔH . However, MFD significantly lowered the T_o (47.7–47.9 °C), T_p (55.7–56.6 °C), and ΔH (3.9–4.1 J/g) for all three potatoes when compared to those of the FD samples (Table 3). T_o , T_p and ΔH were

Table 3

Thermal parameters and texture properties of three potatoes with different treatment^a.

Materials	Treatment	Fracturability (g)	Hardness (g)	T_o (°C)	T_p (°C)	ΔH (J/g)
crumbly potato	FD	137.2 ± 14.5 ^b	209.3 ± 57.1 ^b	60.3 ± 0.1 ^c	64.3 ± 0.1 ^c	12.2 ± 0.1 ^b
	MFD	357.4 ± 83.9 ^a	619.3 ± 102.0 ^a	47.9 ± 0.3 ^a	56.6 ± 0.2 ^a	3.9 ± 0.1 ^a
	FDM	84.2 ± 11 ^b	188.4 ± 31.3 ^b	59.7 ± 0.1 ^b	63.1 ± 0.0 ^b	12.0 ± 0.2 ^b
waxy potato	FD	98.8 ± 13 ^b	204.2 ± 65.3 ^b	60.3 ± 0.1 ^b	63.7 ± 0.0 ^b	15.7 ± 0.9 ^c
	MFD	355.6 ± 94.4 ^a	525.7 ± 122.0 ^a	47.9 ± 0.1 ^a	56.0 ± 0.0 ^a	4.0 ± 0.0 ^a
	FDM	78.2 ± 8.7 ^b	102.0 ± 10.9 ^b	60.4 ± 0.1 ^b	63.6 ± 0.0 ^b	13.0 ± 0.1 ^b
flourey potato	FD	91.2 ± 7.0 ^b	217.6 ± 57.2 ^b	59.6 ± 0.1 ^b	62.9 ± 0.1 ^c	12.8 ± 0.4 ^b
	MFD	307.8 ± 77.4 ^a	466.3 ± 61.5 ^a	47.7 ± 0.2 ^a	55.7 ± 0.0 ^a	4.1 ± 0.1 ^a
	FDM	81.3 ± 11.3 ^b	144.5 ± 14.5 ^b	59.0 ± 0.1 ^c	62.2 ± 0.0 ^b	12.6 ± 0.1 ^b

^a The same small letters indicated there were no significant differences among different treatments for the same variety at the level of $P < 0.05$.

positively related to the content of RS and SDS and negatively correlated to digestion rate (Fig. 1). Colman, Demiate and Schnitzler (2014) found T_o of cassava starch with 18% moisture content was slightly increased after microwave heating for 5 min but decreased after microwave heating for 15 min. This phenomenon was also observed in dry potato starch and starch water suspension treated by microwave (Xie et al., 2013; Kumar et al., 2020). The decreased gelatinization temperature in potatoes treated with MDF suggested that fresh potatoes with high moisture content were easier to be gelatinized when heated directly, the gelatinization of starch and leaching of starch during microwave heating resulted in less intact crystallinity (Xie et al., 2013; Kumar et al., 2020). Zhang, Tian, Liu and Xue (2020) also observed that native potato flour with low gelatinization temperature had lower ΔH and quick melting process compared to the freeze-dried samples. Slight differences of gelatinization properties between in FD and FDM potatoes indicated the microwave heating after freeze-drying did not cause starch gelatinization. Decrease in the degree of gelatinization causes a reduction in starch digestibility *in vitro* and *in vivo* (Rombo et al., 2001).

3.6. X-ray diffraction

Potato starch exhibited B-type polymorph, with typical diffraction peaks at 2 θ 5.6°, 15.1°, 17.1°, 22.2°, and 24.0° (Fig. 3). FDM showed negligible influences on the XRD pattern and crystallinity of starches. While for samples treated with MFD, the diffraction intensity of all peaks became weaker, the diffraction peak at 2 θ 5.6° was even disappeared, and the doublet peaks at 2 θ 22–24° became one weaker single peak (Fig. 3), suggesting the destruction of starch crystal after MFD. This phenomenon was also observed when potato starch was heated with a high microwave (6.63 W/g) (Xu et al., 2019). The decreased intensity of diffraction peaks might be due to the amorphization of the crystalline lamellae (Fan et al., 2016). Correspondingly, the crystallinity of samples treated with MFD were decreased when compared to that of freeze-dried potatoes, while no significant differences were found between those treated with FDM and FD (Fig. 3). It has been found that microwave treatment can either increase, maintain or decrease the relative crystallinity of different starch but the effect of microwave treatment on starch crystallinity was dependent on the moisture content and types of starch (Tao et al., 2020). While compared with other parameters, the crystallinity has negligible contribution for PC1 and PC2 which explain for nearly 90% variations among FD, MFD and FDM potatoes (Fig. 1).

Some studies found microwave treatment can break the double helix structure, contributing to the growth of the amorphous region (Zeng et al., 2016; Wang et al., 2019; Zhou et al., 2019; Tao et al., 2020). The decreased crystallinity in MDF potatoes might also be due to the destruction of starch ordered structure as they also had decreased ΔH (Table 3), which positively correlated to the amount of starch ordered structures (Wang et al., 2018). Tao et al. (2020) proposed that the changed crystallinity caused by microwave might be attributed to the breakage of intra and intermolecular hydrogen bonds rather than the cleavage of the chemical bonds. In this study, the gelatinization of starch caused by microwave dielectric heating may be the main reason for the decreases in crystallinity, as no changes were observed in the samples treated with FDM. During heating in the presence of free water, the water molecules packing the starch double helices vaporized, and a pair of double helices moved into the central channel that was originally occupied by vaporized water molecules, thus the starch polymorph changed from B-type to A-type (Xu et al., 2019). Gelatinization can destroy the double helices in the external starch region and induce the formation of amorphous matrices, a relatively high level of double helical order representing the primary source of resistance to enzyme hydrolysis (García-Rosas et al., 2009), the amorphous matrices may increase the digestion sensitivity (Chung et al., 2006). That's why MFD potatoes showed significantly rapid digestion rate (Fig. 2) and more proportion of RDS (Table 2). Water content was thought to be the crucial factor in affecting starch properties during microwave treatment.

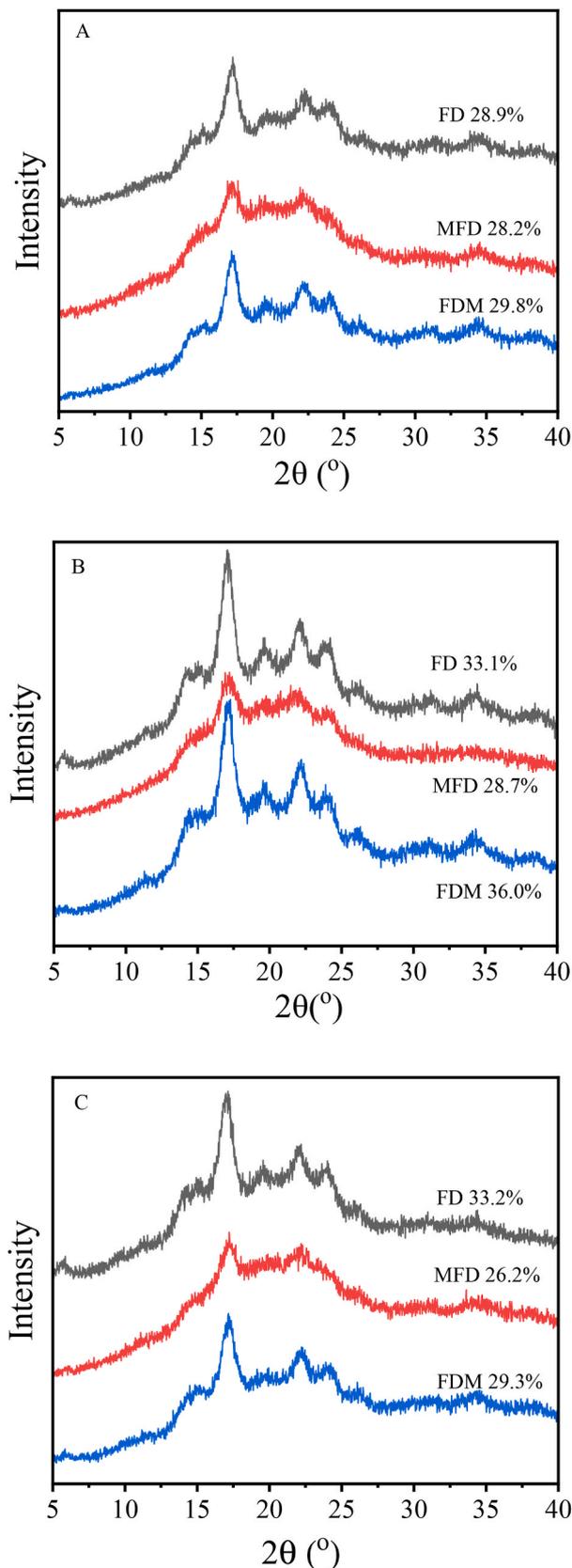


Fig. 3. X-ray diffractograms of three potatoes with different treatments. A: Crummy potato; B: Waxy potato; C: Floury potato. FD: Freeze-drying, MFD: microwave heating before freeze-drying treatment, FDM: microwave heating before freeze-drying treatment.

3.7. GC-MS

In potatoes treated by FD and MFD, three flavor compounds: 2-methyl-butanol, hexanal, and nonanal had been detected. While in potatoes after FDM, the content of nonanal was under detection and the level of hexanal was slightly lowered, but the abundance of 2-methyl-butanol was higher than that in potatoes treated by FD and MFD (Fig. 4). Meantime, some compounds providing baked flavor such as pyrazines, benzeneacetaldehyde, and 2-methyl-propanol were produced after FDM.

Hexanal and nonanal were two raw potato flavor compounds. Hexanal formed by lipoxygenase-initiated reactions, has a green, woody, and fatty flavor (Petersen et al., 1999; Ulrich et al., 2000; Bough et al., 2020), and nonanal has a citrus, green, and potato-like flavor (Petersen et al., 1999; Ulrich et al., 2000). In contrast, methional, benzaldehyde, pyrazines, benzeneacetaldehyde, and 2-methyl-propanol endow a special flavor to baked potato. Among which, pyrazines, the most significant and typical components of baked potato flavor formed through the Maillard reaction (Buttery et al., 1971; Jansky, 2010), have a nutty and roast flavor (Ulrich et al., 2000; Jansky, 2010). Pyrazines were reported to be strongly related to organoleptic quality in baked potatoes (Maga and Sizer, 1973). Other compounds such as methional produced through Strecker degradation reaction also provide a pleasant flavor for cooked and baked potatoes (Ulrich et al., 2000; Jansky, 2010; Bough et al., 2020).

Methional, pyrazines, and 2-methyl-butanol were also found as the flavor compounds of microwaved potatoes in previous studies (Oruna-Concha et al., 2002a; 2002b). However, benzaldehyde and benzeneacetaldehyde were the first time to be detected in FDM treated samples though low in abundance (Fig. 4). Oruna-Concha et al. (2002b) found that flavor compounds in potatoes produced by microwave heating were similar to those prepared with conventionally baking, while the contents are generally slightly lower. The lower or even undetectable level of hexanal and nonanal, and detectable baked flavors in potatoes treated with FDM indicated microwave heating after freeze-drying can reduce the raw potato flavors and give special baked flavors to the cooked potato.

4. Conclusion

Raw potato had a great deal of RS, while few RS will be retained in potatoes after regular cooking. Preserving RS in cooked potatoes as much as possible is meaningful. Potatoes heated with microwave directly (MFD) showed apparently different colors, significantly decreased RS content, and increased digestibility. The gelatinization temperature and the crystallinity of starch after MFD decreased. However, except caused darker color for crumbly potatoes, FDM didn't bring about significant influences on RS content, starch digestibility and other starch properties, which might be because of the limiting gelatinization of starch granules during microwave heating due to the few moisture reserved in FD potatoes. Additionally, FDM potatoes showed special baked flavors. Microwave heating after freeze-drying is a valuable way to reserve RS in cooked potatoes. Water content was thought to be the crucial factor in affecting starch properties during cooking, reducing the moisture in potatoes by freeze-drying before processing seems a potential way to reserve rich RS in crisp, chips, and other processed potatoes.

CRedit authorship contribution statement

Zhangchi Peng: Data curation, Formal analysis, Writing – original draft. **Linrun Cheng:** Investigation, Data curation. **Kaiwei Meng:** Investigation. **Yi Shen:** Resources. **Dianxing Wu:** Supervision, Funding acquisition. **Xiaoli Shu:** Conceptualization, Visualization, Writing – review & editing.

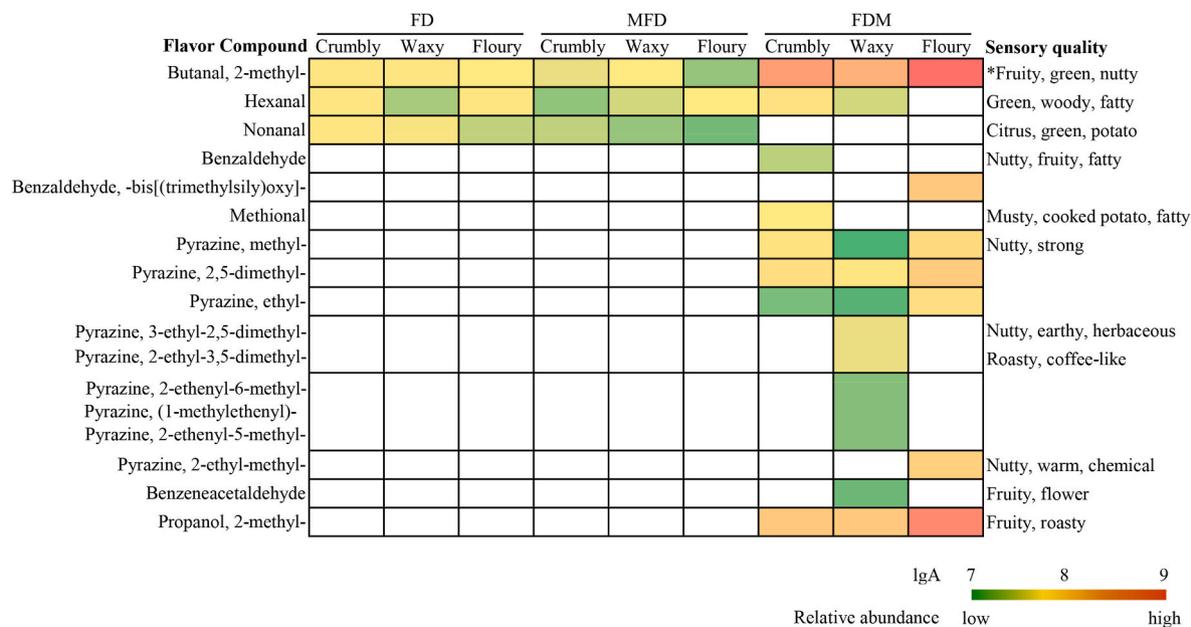


Fig. 4. Heatmap of relative abundance (average peak area). The flavor of each compound was given on the right side of the heatmap. A blank cell means the content of hit compound didn't have significant difference compared with background or the spectrum indicated a low possibility of that compound. FD: Freeze-drying, MFD: microwave heating before freeze-drying treatment, FDM: microwave heating before freeze-drying treatment. * Fruity, green, and nutty is the flavor of 3-methylbutanal. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Declaration of competing interest

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.

Data availability

No data was used for the research described in the article.

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