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# Effects of different roasting conditions on sugars profile, volatile compounds, carotenoids and antioxidant activities of orange-fleshed sweet potato

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#### ABSTRACT

The effects of different roasting conditions (180 °C/70 min (T180), 210 °C/50 min (T210), and 240 °C/30 min (T240) on the qualities of orange-fleshed sweet potato (OFSP) were explored. Changes in sugars and carotenoids were analyzed by GC–MS and LC-MS/MS, and volatile compounds were characterized by GC × GC/TOF-MS. The antioxidant activities (DPPH• and ABTS•†) and bioaccessibility of  $\beta$ -carotene after in *vitro* digestion were also evaluated. Results showed that sugar content increased with roasting temperature, with T240 showing the highest sugar content (817.53 mg/g). The greatest variety of sugar species was identified in T180, with maltose (53.51 %) and sucrose (25.70 %) being the predominant sugars. In addition, T210 produced the largest number of volatile compounds, with vanillin being key flavor compound. Regarding the antioxidant activities and bioaccessibility in *vitro* simulated digestion, their capacity was T180 > T210 > T240. In comparison, T210 appears to be the optimal roasting condition, offering a balanced sweetness profile, enhanced flavor complexity, and optimal retention of carotenoids.

#### 1. Introduction

Sweet potato (Ipomoea batatas L.) is one of the most important, versatile, and under-exploited food crops in the world, and plays an important role in global food security (Dung, Giau, Van Hao, Minh, & Thuy, 2024). The orange-fleshed sweet potato (OFSP) is a variety of sweet potato that serves as an excellent source of carbohydrates, enriched with essential nutrients including vitamins, amino acids, and minerals, particularly carotenoids (Olaniran et al., 2024). This makes OFSP beneficial for combating vitamin A deficiency, particularly in children and pregnant women (Alam, Sams, Rana, Akhtaruzzaman, & Islam, 2020). Additionally, high carotenoid intake may help prevent aging, cardiovascular diseases, and certain cancers (Bakac, Percin, Gunes-Bayir, & Dadak, 2023; Gebregziabher et al., 2023; Pan, Sun, Zhang, Guo, & Liao, 2019). In the current processing methods of OFSP, roasting is one of the most commonly used preparation methods, valued for its simplicity, cost-effectiveness, and accessibility. Roasting enhances the texture and flavor profile of OFSP, improving its overall sensory qualities, which contributes to its popularity (Jati, Darmoatmodjo, Suseno, Ristiarini, & Wibowo, 2022; Nicoletto, Vianello, & Sambo,

2018). Moreover, extensive research has demonstrated that roasting improves the sensory attributes, digestibility, and palatability of various food products, such as tea, coffee, and oats, through the Maillard reaction and caramelization processes, which induce favorable structural modifications (Freitas et al., 2023; Schlörmann et al., 2020).

Most of the current studies on roasting sweet potatoes have focused on starch content and structure, total sugars, total carotenoids, and antioxidant activity (Kourouma, Mu, Zhang, & Sun, 2019). However, few studies have reported variations in other carotenoid components, such as the specific carotenoid profiles in different roasting processes. In addition, changes in starch and sugar content after roasting have been reported, with a decrease in starch, especially maltose (Zhang et al., 2023), while there is less understanding of the dynamics of other key metabolites. Recent study showed that roasting increased the types and concentrations of volatile compounds in sweet potatoes, which was higher than that in steamed sweet potatoes (Yao, Zhang, Jia, Deng, & Wang, 2023). Given that the roasting process plays a crucial role in improving the flavor and quality of sweet potatoes, further research was needed to systematically assess the dynamic changes in chemical composition and flavor profile of OFSP during roasting. The

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comprehensive investigation of these changes across different roasting methods is crucial for better understanding the underlying mechanisms of flavor development and nutritional enhancement.

In this study, to elucidate their dynamics and to further understand the mechanism of flavor formation in roasted OFSP, the effects of three roasting conditions ((180 °C/70 min (T180), 210 °C/50 min (T210), and 240 °C/30 min (T240)) on sugars, carotenoids, and volatile compounds in OFSP were investigated based on traditional analytical and metabolomics techniques. In addition, the antioxidant capacity during digestion of different roasted OFSPs also explored, which may help consumers better understand its nutritional and bioactive qualities. Understanding the chemical profiles associated with roasting methods may provide new dietary health insights into the nutritional and bioactive compounds of sweet potatoes, thereby contributing to technological advances in the sweet potato processing industry.

#### 2. Materials and methods

#### 2.1. Materials

Orange-fleshed sweet potatoes (Yantian Potato No. 25: 72.8 % water, 24.3 % carbohydrates, 3.0 % fiber, 1.7 % protein, 0.3 % lipid) were purchased from Hebei Xiong'an Potato Big Brother Honey Potato Co. and produced in Yantai City, Shandong Province.

#### 2.2. Roasted conditions and sample preparation

Fresh medium-fruited long OFSP of medium size (20–27 cm), weight (250–350 g), and similar phenotype were selected, keeping the sweet potato skin as intact as possible. After pre-testing, the roasting temperatures and times were determined to be  $180\,^{\circ}\text{C}/70$  min,  $210\,^{\circ}\text{C}/50$  min, and  $240\,^{\circ}\text{C}/30$  min. OFSP was roasted in an oven (CRWF42NE, Vesta, China) and sampled:  $180\,^{\circ}\text{C}/35$  min (T180M),  $180\,^{\circ}\text{C}/70$  min (T180),  $210\,^{\circ}\text{C}/25$  min (T210M),  $210\,^{\circ}\text{C}/50$  min (T210),  $240\,^{\circ}\text{C}/15$  min (T240M) and  $240\,^{\circ}\text{C}/30$  min (T240). The raw OFSP (T0) was used as a control. Each sample was frozen in liquid nitrogen for subsequent tests.

# 2.3. Determination of the color of OFSP flesh

According to the method from the previous methodology with some modifications, the color parameters of different OFSP samples were tested using a colorimeter (ColorQuest XE, HunterLab, USA)(Zhang et al., 2023). Before measuring the samples, the colorimeter was calibrated using a black and white standard plate based on three color parameters: L (lightness), a\* (redness and greenness), and b\* (yellowness and blueness).

#### 2.4. Sugars fractions determination

#### 2.4.1. Sugars extraction

Sugars were extracted using a modified method based on the previous methodology (Zhang et al., 2020). The freeze-dried samples were crushed using a mixer mill (MM 400, Retsch) with a zirconia bead for 1.5 min at 30 Hz. 20 mg of powder was diluted to a 500  $\mu L$  with methanol: isopropanol: water (3:3:2, v/v/v), then vortexed for 3 min and ultrasound for 30 min. After that, the extract was centrifuged at 12,000 rpm under 4 °C for 3 min to collect supernatant. Then 50  $\mu L$  of the supernatant was mixed with 20  $\mu L$  internal standard (1000  $\mu g/mL$ ) and evaporated under a nitrogen gas stream. Finally, The evaporated sample was transferred to the lyophilizer for freeze-drying. The residue was used for the further derivatization.

The derivatization method was as follows (Zheng, Zhang, Quan, Zheng, & Xi, 2016): the sample was mixed with a 100  $\mu L$  solution of methoxyamine hydrochloride in pyridine (15 mg/mL). The mixture was incubated at 37 °C for 2 h. Then 100  $\mu L$  of BSTFA was added into the mixture and kept at 37 °C for 30 min after vortex-mixing. The mixture

was analyzed by GC-MS after diluting to an appropriate concentration.

#### 2.4.2. Quantification of sugars

Agilent 8890 gas chromatograph coupled to a 5977B mass spectrometer with a DB-5MS column (30 m length  $\times$  0.25 mm i.d.  $\times$  0.25 µm film thickness, J&W Scientific, USA) was employed for GC–MS analysis of sugars. Helium was used as carrier gas at a 1 mL/min flow rate. Injections were made in the split mode with a split ratio of 5:1 and the injection volume was 1 µL. The oven temperature was set to 320 °C and held at this temperature for 5.5 min. All samples were analyzed in selective ion monitoring mode. Temperatures of ion source and transmission line were 230 °C and 280 °C, respectively (Sun, Wang, Xie, & Su, 2016).

# 2.5. Volatile compounds determination

Volatile compounds of OFSP were analyzed using GC  $\times$  GC/TOF MS. A stock solution with 10 mg/L of n-Hexyl-d13 was prepared in 50 % ethanol, and stored at 4 °Crefrigerator. A stock solution with 1 mg/L of Saturated Alksnes was prepared in N-Hexane and stored at 4 °C refrigerator.1 g of sample and 10 µL of the internal standard were added into a 20 mL bottle, then prepared samples were incubated at 60 °C for 10 min. Before the adsorption of the sample, the SPME extractor head was aged at 270 °C for 10 min; the aged SPME extractor head was transferred to the incubation chamber, and the sample was adsorbed at 60  $^{\circ}\text{C}$  for 15 min. After adsorption, the SPME extractor head was transferred to the incubation chamber, and the sample was adsorbed for 15 min; after adsorption, the SPME extractor head was transferred to the incubation chamber, and the SPME extractor head was transferred to the incubation chamber. After the adsorption, the SPME extractor head was transferred to the GC injection port and desorbed for 5 min at 250 °C; after sample injection, the SPME extractor head was aged for 10 min at 270  $^{\circ}\text{C}.$  10  $\mu L$ of n-alkanes were taken into a 20 mL headspace injection vial and then extracted and injected into the injection vial by incubation (Corrales et al., 2017; Hou, Mu, Ma, & Blecker, 2019).

Qualitative analysis of volatile components was based on comparisons of the National Institute of Standards and Technology (NIST08) mass spectral libraries and by matching the retention index (RI) values with those reported in the literature. To enable data of different magnitudes to be compared, the total peak area normalization or internal standard normalization was performed on the raw data (Feizi, Hashemi-Nasab, Golpelichi, Saburouh, & Parastar, 2021). This project uses internal standards for normalization. Semiquantitative data of the volatile compounds were quantified by calculating the peak area.

# 2.6. Carotenoids fractions determination

#### 2.6.1. Carotenoids extraction

The sample was freeze-dried, ground into powder (30 Hz, 1.5 min), and stored at  $-80~^{\circ}\text{C}$  until needed. 50 mg powder was weighted and extracted with 0.5 mL mixed solution of n-hexane: acetone: ethanol (1:1:1, v/v/v). The extract was vortexed for 20 min at room temperature. The supernatants were collected after centrifuged at 12000 r/min for 5 min at 4  $^{\circ}\text{C}$ . Then, the collected supernatant was evaporated to dryness and redissolved in 100  $\mu$ L dichloromethane. The prepared solution was filtered through a 0.22  $\mu$ m membrane filter for further LC-MS/MS analysis (Inbaraj et al., 2008; Rivera, Christou, & Canela-Garayoa, 2013).

#### 2.6.2. Quantification of carotenoids

The sample extracts were analyzed using a UPLC-APCI-MS/MS system (UPLC, ExionLCTM AD, https://sciex.com.cn/; MS, Applied Biosystems 6500 Triple Quadrupole, https://sciex.com.cn/) with YMC C30 column (3  $\mu m$ , 100 mm  $\times$  2.0 mm). The solvents used in this experiment were acetonitrile (1:3, v/v) with 0.01 % BHT and 0.1 % formic acid (A) and methyl tert-butyl ether with 0.01 % BHT (B). Additionally, the

gradient program began at 0 % B (0–3 min), increased to 70 % B (3–5 min), then rose to 95 % B (5–9 min), and ended at 0 % B (10–11 min). The column temperature was 28  $^{\circ}$ C, and the flow rate was 0.8 mL/min (Petry & Mercadante, 2017).

APCI-MS/MS Conditions: An API 6500 Q TRAP LC/MS/MS System, equipped with an APCI Turbo Ion-Spray interface, was operated in positive ion mode and controlled by Analyst 1.6.3 software (AB Sciex). The APCI source operation parameters were as follows: ion source, APCI+; source temperature, 350 °C; curtain gas (CUR), 25.0 psi; and collision gas (CAD), medium. Declustering potential (DP) and collision energy (CE) for individual MRM transitions were performed with further DP and CE optimization. A specific set of MRM transitions was monitored for each period according to the carotenoids eluted within each period. The integrated peak area of each carotenoid detected in the samples was substituted into the linear equations of standard curves for content calculation.

# 2.7. Determination of antioxidant capacity and bioaccessibility after in vitro simulated digestion

#### 2.7.1. $\beta$ -carotene during in vitro digestion

The in *vitro* digestion experiments were carried out by referring to the method of Saracila and Li et al. with slight modifications (Li, Xiao, Tu, Yu, & Niu, 2021; Saracila, Untea, Oancea, Varzaru, & Vlaicu, 2024; Sun et al., 2018). 1 g of powdered dried samples and the appropriate amount of buffer were mixed in pepsin solution, then adjusted to the pH 2.5 with 1 mol/L HCl solution for 60 min of simulated gastric digestion. Then the pH was adjusted to 6.8 by adding 1 mol/L NaHCO $_3$  solution, and the enzyme mixtures (porcine pancreatic enzyme and saccharase) with a ratio of 20:1 were added and kept in a water bath at 37 °C for 180 min. Then, 1 mL of the digested mixtures was obtained for inactivating the enzyme for the determination of antioxidant capacity and bioaccessibility of  $\beta$ -carotene.

The  $\beta$ -carotene was extracted by ultrasound-assisted method (Wu, Zhang, Ye, & Yu, 2020). 1 mL of the digested mixture was weighed and 10 mL of n-hexane/petroleum ether/ethanol (6:3:1, v/v). After sealing and homogenization, the mixture was treated with ultrasound for 25 min, followed by incubation in an oscillating water bath at 50 °C in the dark for 1 h. Afterward, the extract was centrifuged at 1409 g for 10 min, and the supernatant was collected for analysis. The extraction process was repeated three times. The  $\beta$ -carotene concentration was determined using the calibration curve (y = 19.213x + 0.2072, R<sup>2</sup> = 0.9959). Each sample was analyzed in triplicate, and the entire experiment was conducted under light protection.

# 2.7.2. Ferric-reducing antioxidant power (FRAP) assay

The FRAP assay was performed with slight modifications based on the method of previous methodology (Yan et al., 2018). The FRAP reagents were prepared by mixing sodium acetate buffer (300 mmol/L, pH 3.6), TPTZ solution (10 mmol/L, 40 mmol/L HCl), and FeCl $_3\cdot$ 6H $_2$ O solution (20 mmol/L) in a ratio of 10:1:1. The reagent (3 mL) was preheated to 37 °C, then mixed with 1 mL of the digested sample. An equal volume of distilled water was used as the blank. Absorbance was measured at 593 nm after incubating in a water bath for 30 min. A calibration curve (y = 3.1479x + 0.0482, R² = 0.9967) was constructed using Fe(II) solutions (10–100 µmol/L FeSO<sub>4</sub>·7H $_2$ O), and the results were expressed as mmol/L Fe²+/g dry weight.

# 2.7.3. DPPH• radical scavenging assay

DPPH• was prepared with a slight modification based on the previous methodology (Feng et al., 2023). 25 mg of DPPH was dissolved in 1 L of anhydrous ethanol to form a DPPH-ethanol solution with a concentration of  $6.5 \times 10^{-5}$  mol/L. Then, 1 mL of digested mixtures and DPPH-ethanol solution were added into a test tube and shaken well, and the reaction was carried out at 25 °C for 30 min in the dark. Absorbance was measured at 517 nm by UV spectrophotometer and DPPH• scavenging

activity (%) was calculated according to the following equation (1):

Scavenging activity(%) = 
$$\left[1 - \left(\frac{A_{sample}}{A_0}\right)\right] \times 100$$
 (1)

where  $A_{sample}$  and  $A_0$  are absorbance measurements at 515 nm for the sample and control, respectively. The  $IC_{50}$  value is the effective  $\beta$ -carotene concentration for scavenging 50 % of DPPH free radicals.

# 2.7.4. ABTS•+ radical scavenging assay

ABTS•<sup>+</sup> radical scavenging activity was determined according to the slightly modified method of Feng and Liang et al. (Feng et al., 2023; Liang et al., 2012). The ABTS•<sup>+</sup> working solution was prepared by mixing 4 mL of 7.4 mmol/L ABTS solution with 14.3 mL of 2.6 mmol/L potassium persulfate solution and allowed to stand in the dark at 25 °C for 12–16 h. The resulting solution was diluted with anhydrous ethanol to achieve an absorbance of 0.7  $\pm$  0.02 at 734 nm. Next, 50  $\mu$ L of the digested mixture was combined with 4.95 mL of diluted ABTS•<sup>+</sup> working solution, and the reaction was conducted at 25 °C for 30 min in the dark. Absorbance was measured at 734 nm. Anhydrous ethanol (50  $\mu$ L) was used as the blank control. The ABTS•<sup>+</sup> radical scavenging activity (%) was calculated using equation (2):

where  $A_{sample}$  and  $A_0$  are the absorbance values at 734 nm for the sample and control, respectively. The  $IC_{50}$  value is the effective  $\beta$ -carotene concentration for scavenging 50 % of the ABTS•<sup>+</sup> free radicals.

#### 2.7.5. Determination of bioaccessibility

Bioaccessibility was determined according to a slightly modified version of the method of Jin et al. (Jin et al., 2023). The bioaccessibility of carotenoids in OFSP as compared to the initial content of carotenoids in undigested OFSP, and the bioaccessibility was according to the following equation (3):

$$M = C_X/C_T \times 100 \tag{3}$$

where M is the bioaccessibility, %;  $C_X$  is the concentration of  $\beta$ -carotene in the digested mixtures,  $\mu g/g$ ;  $C_T$  is the initial  $\beta$ -carotene concentration in the sample,  $\mu g/g$ . Refer to section 2.7.1 for the extraction procedure of  $\beta$ -carotene from  $C_X$  and  $C_T$ .

# 2.8. Statistical analysis

To ensure the reliability of the experimental results, each group was assessed in three replications. All data were analyzed using SPSS statistical 27 and significance was determined by LSD post-hoc test at p < 0.05

# 3. Results and discussion

# 3.1. Appearance properties

OFSP was subjected to high-temperature roasting, resulting in the development of a vibrant orange flesh that exhibited an intensified browning response as the temperature increased, this phenomenon was particularly evident at higher roasting temperatures (Fig. 1A). The color and sheen of both the flesh and skin of the OFSP were significantly influenced by the roasting conditions. T180 consistently displayed a bright flesh color, smooth skin texture, and vibrant skin hue. T210 showcased charred brown spots on the surface of the flesh, enhancing its visual appeal and palatability. T240 demonstrated pronounced scorching discoloration of both pulp and rind, accompanied by the release of molasses-like material from the pulp's surface. All three roasting conditions had a substantial impact on improving or altering the visual

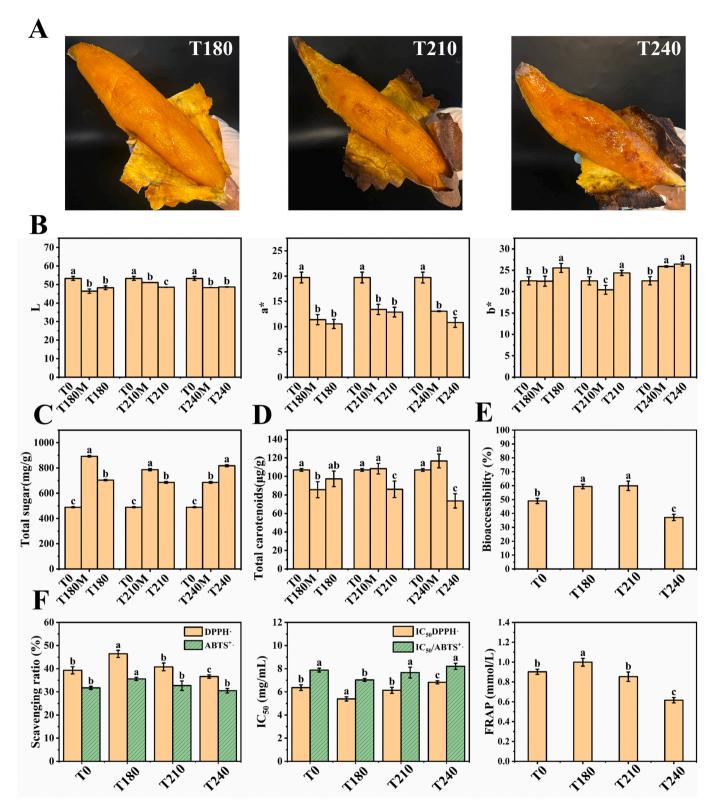


Fig. 1. Changes in OFSP under different roasting conditions. (A) Morphological observation of ripened OFSP. (B) Values of three color parameters (L,  $a^*$ ,  $b^*$ ) of OFSP detected by a colorimeter. (C) Total sugar content (mg/g) of OFSP during roasting. (D) Total carotenoid content (µg/g) of OFSP during roasting. (E) Bioaccessibility of roasted OFSP (F) Antioxidant capacity (DPPH• and ABTS • scavenging, IC<sub>50</sub>, FRAP reduction capacity) of roasted OFSP after simulated digestion. Different letters indicate significant differences (P < 0.05).

quality characteristics of OFSP.

The color parameters (L,  $a^*$ , and  $b^*$ ) of the raw and roasted samples are illustrated in Fig. 1B. The roasted samples exhibited a gradual decline in L and  $a^*$  values, which were significantly lower than those of

the raw samples under the three distinct roasting conditions. In contrast, the b\* values showed an increase after the roasting process. Among all conditions, T240 had the lowest L value and highest b\* value, while T180 displayed the lowest a\* value. The color change during roasting

may be caused by the color of the Maillard reaction and caramelization reaction products (López et al., 2010). Previous studies have found that the decrease in the L-value of the samples during roasting was caused by the brown pigment, which is a product of the Maillard reaction (Koriyama, Teranaka, Tsuchida, & Kasai, 2022). Simultaneously, the enzymatic reactions occurring during the baking process promote the hydrolysis of starch in OFSP, thereby diminishing the product's brightness (Punia Bangar, Ashogbon, Singh, Chaudhary, & Whiteside, 2022). In conclusion, higher roasting temperatures lead to more intense chemical reactions in OFSP, such as the Maillard and caramelization reactions, resulting in increased starch hydrolysis. These alterations contribute to a darker coloration and reduced brightness of the samples, thereby enhancing their visual appeal.

#### 3.2. Sugar content

#### 3.2.1. Total sugar content

Significant variations were observed in the total sugar contents of all samples, which depended on both the duration and temperature of roasting (Fig. 1C). Specifically, an increase in roasting temperature led to a rise in sugar content from 40.14 % to 82.50 %. The sugar content was measured at 489.15 mg/g for T0. The results indicated that the total sugar content in T180M and T210M reached their respective maxima of 892.68 mg/g and 789.60 mg/g at 180  $^{\circ}$ C and 210  $^{\circ}$ C, respectively. Further roasting at these temperatures led to a decrease in sugar content to 703.56 mg/g for T180 and 685.51 mg/g for T210. However, at 240 °C, the total sugar content of T240M was lower at 763.86 mg/g compared to T180M and T210M. Notably, during subsequent roasting, the sugar content in T240 increased continuously to 817.53 mg/g, ultimately resulting in a higher final sugar content than both T180 and T210. Sharma et al. found that the decrease in sugar content may occur because of the formation of Maillard reaction products, which are formed by the reaction between sugars, amino acids, and proteins (Sharma et al., 2015). Marasinghege et al. found that the rate of sucrose degradation in sugarcane juice increased with heating surface temperatures (<2.5 % at 168 °C) (Marasinghege et al., 2022). Hou et al. found that a large amount of starch was degraded to sugar, especially converted to maltose, during the heat treatment of sweet potato (Hou et al., 2019). These findings suggest that the sugar content in OFSP undergoes complex changes during roasting due to temperature and time, which may be accompanied by Maillard reaction and sugar conversion degradation, among others.

#### 3.2.2. Soluble sugar content

Principal Component Analysis (PCA) based on sugar levels revealed various groups, with significant differences between them. PC1 and PC2 accounted for 41.78 % and 24.22 % of the total variance, respectively, contributing to a combined explanation of 66 % (Fig. 2 A<sub>1</sub>). All samples except T0 were successfully segregated and clustered into distinct groups, indicating significant disparities in sugar profiles. According to the molecular structure of carbohydrate compounds, sugars were classified into monosaccharides and disaccharides (Niaz, Khan, & Shah, 2020). During roasting, a decrease in monosaccharide content and an increase in disaccharide content were observed. The highest concentration of monosaccharides was observed at T0, with the lowest in T240. Whereas T180M and T240 had the highest concentration of disaccharides, and T0 had the lowest (Fig. 2  $B_1$ ). As depicted in Fig. 2  $C_1$ , a total of 25 distinct sugars were identified, comprising 20 monosaccharides and 5 disaccharides. To contained 23 kinds of sugars, with counts reaching up to 25 for T180M, T180, and T210M followed by counts of 24 for T210, T240M, and T240. Notably, D-xylulose was not detectable at T0, T210, T240 and T240M. The absence of 2-deoxy-Dglucose was exclusively observed at T0.

Eight soluble sugars (rhamnose, arabinose, galactose, mannose, glucose, fructose, sucrose, and maltose) across all samples were illustrated in Fig. 2  $C_1$ , and significant variations in soluble sugar content

were observed. Studies found that soluble sugar content was directly related to the sweetness of roasted sweet potatoes, affecting sweet potatoes' viscosity, texture, flavor, and other taste indexes (Ando, Yasuda, & Hisaka, 2018). Glucose and fructose were the primary contributors to sweetness (Xiao et al., 2024). In this experiment, galactose content exhibited a decline with increasing temperature and duration of roasting, reaching its highest level in the T0 (1.68 mg/g). Glucose, sucrose, and fructose were identified as the primary sugars present in the T0 at concentrations of 192.70 mg/g (39.39 %), 177.68 mg/g (35.71 %), and 111.08 mg/g (22.71 %) respectively. In T180, T210, and T240, the glucose, sucrose and fructose accounted for 5.96 %  $\sim$  25.51 %, 7.26 %  $\sim$  31.26 %, and 3.00 %  $\sim$  20.90 % respectively (Fig. 2 C<sub>1</sub>). Both fructose and glucose contents showed a similar trend during the roasting process (Fig. 2 C<sub>1</sub>), while the sucrose content exhibited relatively stable levels during the mid-and late-roasting stages, contrasting with other soluble sugars. The maltose content of the T0 was only 2.38 mg/g (0.49 %), while a significant increase in maltose content was observed after the roasting process. During the roasting process, the content of maltose in T180, T210, and T240 was increased by 53.10 %, 43.71 %, and 66.63 %, respectively, and their final contents were 373.62 mg/g, 299.64 mg/g, 544.75 mg/g. These findings suggest that the roasting process significantly promotes the formation of maltose compared to other sugar fractions. Simultaneously, higher temperatures and short periods for brief durations are more conducive to the generation and accumulation of maltose. The aforementioned observation aligns with the findings reported by Chan et al., furthermore, they revealed that a significant increase in maltose levels during the processing procedure substantially enhanced the sweetness and overall acceptability of roasted sweet potatoes (Chan et al., 2014). This was consistent with previous findings that maltose was the major sugar in cooked sweet potatoes (Nicoletto et al., 2018). On the other hand, Grabowski et al. discovered a significant presence of maltose in roasted sweet potatoes, resulting from the enzymatic hydrolysis of starch catalyzed by amylase during the roasting process (Grabowski, Truong, & Daubert, 2008). The hydrolysis of starch during roasting has been reported to be catalyzed by three types of amylases:  $\alpha$ -amylase,  $\beta$ -amylase, and starch phosphorylase, among these enzymes,  $\beta$ -amylase was found to be the most abundant, constituting approximately 5 % of the total protein content in sweet potato roots (Banda et al., 2021). It was also found that the above three amylases would hydrolyze the non-reducing α-1,4-glycosidic bond of starch to produce glucose or maltose during roasting, and this hydrolysis led to a large amount of maltose production (Liu et al., 2023). The roasting process of OFSP can be considered as a complex phenomenon.

OFSP contains a large amount of starch, which undergoes hydrolysis during the roasting process involving the conversion of starch into soluble sugars and leading to a significant increase in soluble sugars, especially disaccharides such as maltose and cellobiose. This process was affected by changes in temperature and time, resulting in significant differences in the composition of the various sugar fractions in OFSP. Moreover, compared to raw OFSP, the roasted OFSP identified an expanded range of sugar types. However, the T240 sample exhibited the highest sugar content, the levels of glucose and fructose as well as the identified types of sugars were comparatively lower than those in the other roasted samples. Consequently, it can be inferred that the sweetness profile of T240 would be less robust compared to that of T180 and T210.

#### 3.3. Volatile metabolic compounds

#### 3.3.1. Overview of volatile metabolic compounds

Fig. 5 presents comprehensive information on the volatile compounds and their relative contents, a total of 86 volatile compounds were identified across all samples. The roasted OFSP exhibited the highest number of identified compounds in T210 (659), followed by T180 (635) and T240 (604) (Fig. 3A). Fig. 3C and D illustrated the distribution of compound content within each sample group, presenting

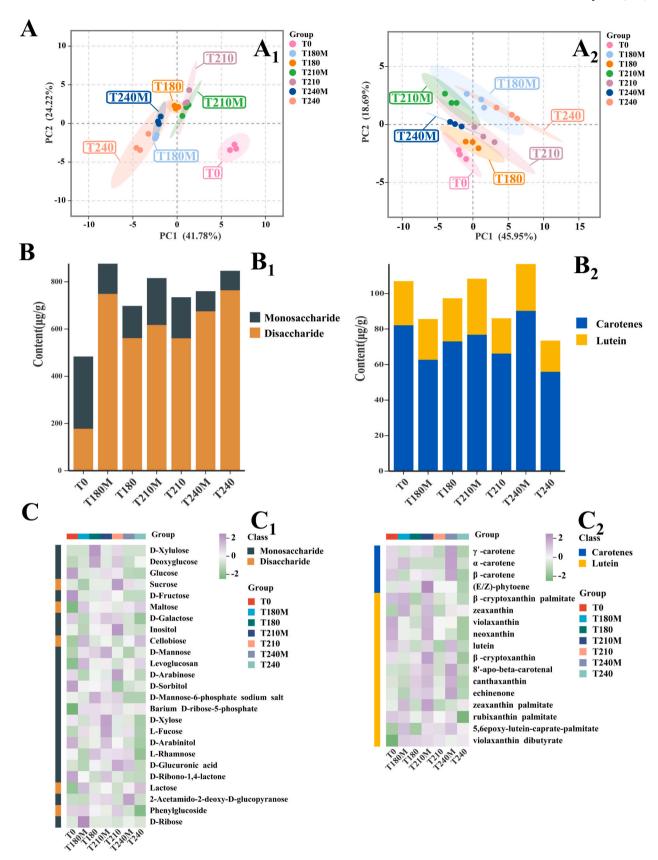


Fig. 2. Changes in sugars and carotenoids in lower OFSP during different roasting treatments. (A) Plot of PCA scores. (B) Class statistics of compounds. (C) Clustering heat map of compounds. (A<sub>1</sub>, B<sub>1</sub>, and C<sub>1</sub> are sugar compounds; A<sub>2</sub>, B<sub>2</sub>, and C<sub>2</sub> are carotenoid.)

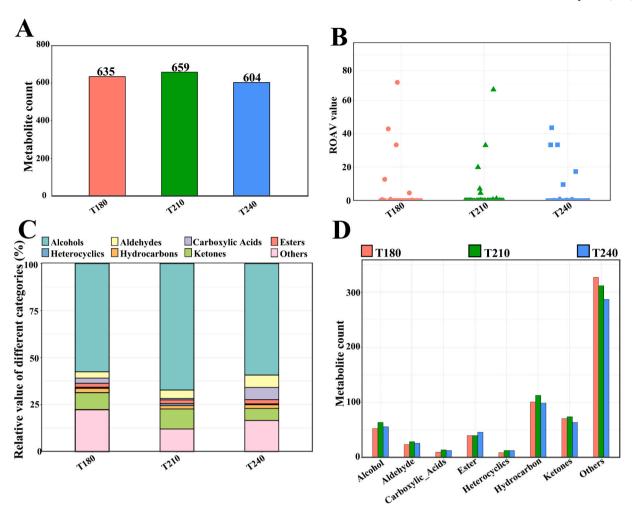


Fig. 3. Overview of volatile compounds in OFSP under different roasting treatments. (A) Plot of number of substance identifications. (B) Scatter plot of ROAV odor activity values. (C) The stacked plot of relative content of substance species. (D) Statistical bar chart of the number of substance species. Simulated post-digestive antioxidant capacity of OFSP with different roasting treatments.

a comprehensive summary of major categories including alcohols, aldehydes, carboxylic acids, esters, heterocyclic compounds, and hydrocarbons. Notably, alcohol compounds were found to be the most abundant in the roasted OFSP samples with significant variations observed among different groups.

In Fig. 4A, Wayne illustrates the count of overlapping substances, where the intersecting portion represents the number of substances concurrently identified by multiple sample groups, while the nonintersecting portion signifies the count of substances exclusively identified by their respective sample group. The number of specific flavor compounds identified in T180, T210, and T240 were 194, 198, and 150 respectively. A total of 305 flavor compounds were identified across all three sample groups; among these, 76, 60, and 80 were jointly identified by T180 vs T210, T180 vs T240, and T210 vs T240, respectively. Substance names, maximum/minimum concentration ranges, as well as sensory flavors for the volatile compounds detected were assigned using the Odor and Flavordb databases. The Flavourdb database was utilized to assess and compare the organoleptic flavors of compounds in OFSP roasted under varying roasting conditions on a frequency scale ranging from 1 to 5. Among the three groups of roasted samples, sweet, green, and fruity flavors were most prominent (Fig. 4B).

To evaluate the contribution of each volatile component to the overall flavor, Relative Odor Activity Value (ROAV) was utilized, with a compound considered as a key flavor compound when  $ROAV \ge 1$  (Zhu, Chen, Chen, Chen, & Deng, 2020). Fig. 3B presents a scatter plot illustrating the ROAV odor activity values for the roasted samples. Notably,

aldehydes exhibited the highest ROAV values among all analyzed compounds. This finding aligned with the results that aldehydes were the predominant compounds in different sweet potato flavors, followed by alcohols, ketones, and terpenes (Gu et al., 2023), and Zhao et al. found aldehydes were considered to be key flavor compounds in many foods because of their low taste thresholds (Zhao et al., 2020). In addition, Li et al. found that the higher the heating temperature, the greater the formation and accumulation of aldehydes (Li et al., 2018; Liu et al., 2020). While in this study, T180 demonstrated the most pronounced accumulation of aldehydes. Furthermore, the formation of 3-methyl butyraldehyde, 2-methyl butyraldehyde, and 2-methyl propionaldehyde in the samples was thought to be associated with nonenzymatic, heat-induced, Strecker degradation of the aminosugar and reducing sugar fractions, which was often perceived as a malty, chocolate-like flavor (Smit, Engels, & Smit, 2009).

Among these groups, compounds with ROAV values  $\geq 1$  included 2-nonenal, vanillin, 2-methyl-butanal, 2-pentyl-furan, heptanal, and 2-undecane (Fig. 5). 2-nonenal is a fatty acid degradation product associated with the flavor of green leaves or fats, providing a fruity or fatty odor (Gigot, Ongena, Fauconnier, Wathelet Paul, & Jardin Du, 2010). Furan is thought to be the result of a Maillard reaction, 2-Pentyl-furan with a green and fruity odor (Krishnamurthy, Smouse, Mookherjee, Reddy, & Chang, 1967). Notably, 2-nonenal exhibited the highest ROAV value both at T180 and T240, whereas vanillin displayed the highest ROAV value at T210 a very sweet flavor (Zeng et al., 2024). In addition, maltol has previously been shown to be an important component in the

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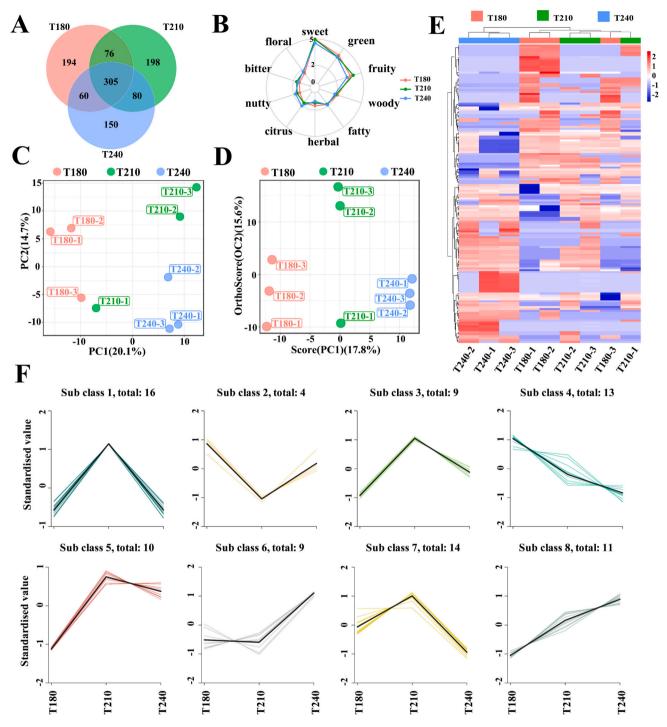


Fig. 4. Analysis of volatile compounds in OFSP under different roasting treatments. (A) Wayne's diagram of the number of substances overlaps. (B) Radar plot of flavor characterization. (C) PCA score plot. (D) OPLS-DA score plot ( $R^2X = 0.334$ ;  $R^2Y = 0.999$ ;  $Q^2 = 0.603$ ). (E) Statistical bar graph of the number of substance species. (E) Heat map of all metabolites. The redder the color the higher the relative content, and the bluer the lower the relative content. (F) K-means analysis of all metabolites.

flavor of roasted sweet potatoes. In summary, differences in the content of these compounds in the roasted OFSP give it a different flavor profile. The flavor profiles observed in roasted OFSP were generally similar encompassing predominantly sweet notes along with green and fruity nuances.

#### 3.3.2. Differential compounds

Differential compounds were identified using fold change and VIP analysis, aiming to comprehensively elucidate the organoleptic

properties associated with roasted OFSP. The screening criteria were set as VIP  $\geq 1$  and p < 0.05, ensuring a rigorous selection of significant compounds. The screening results are presented in Fig. 6, revealing the identification of 44 compounds across all roasted samples (Fig. 6A). The PCA of the differential compounds is depicted in Fig. 4C. PC1 and PC2 accounted for 20.1 % and 14.7 % of the variance, respectively, contributing to a combined explanation of 34.8 % of the total variance. The heat map resulting from Hierarchical Cluster Analysis (HCA) on volatile compounds (Fig. 4E) further confirmed substantial differences

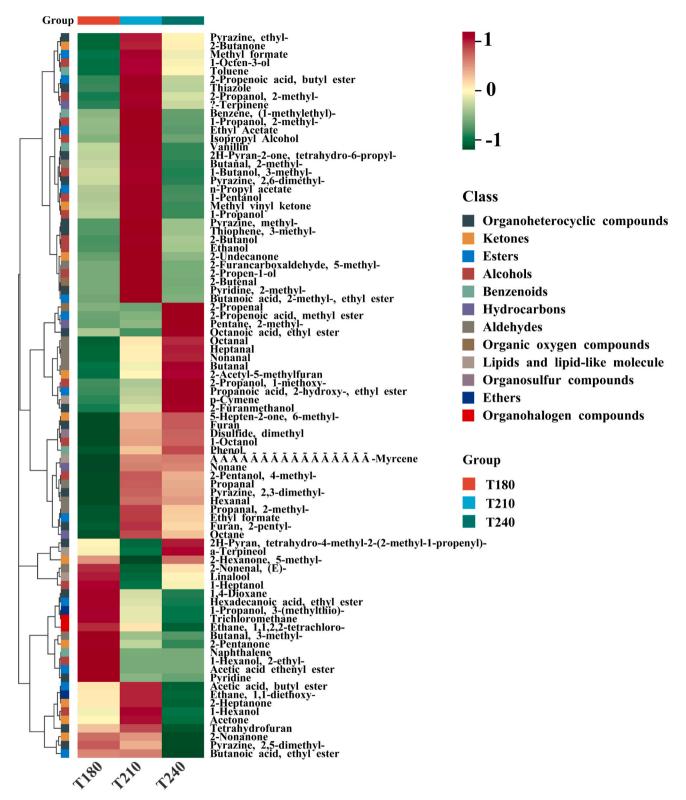
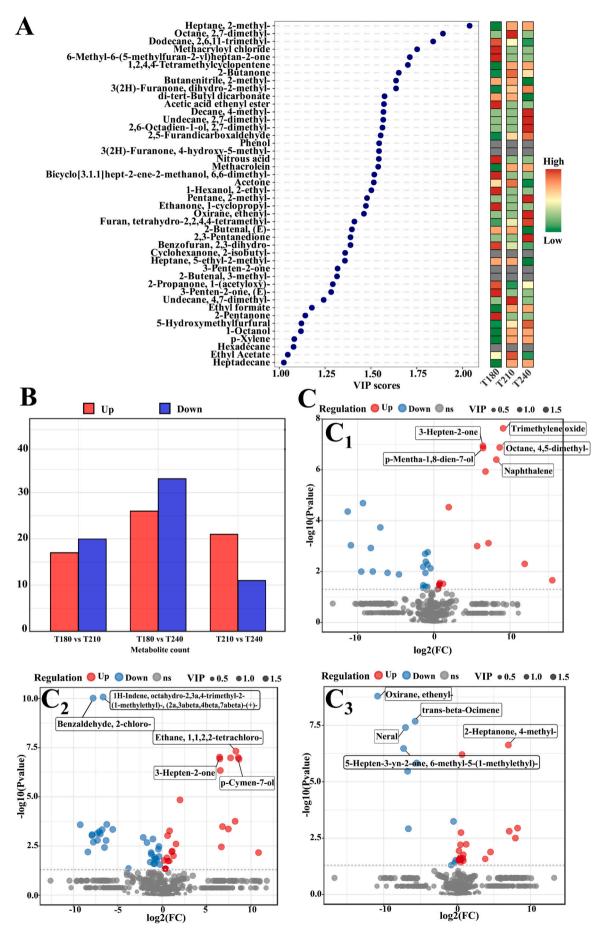


Fig. 5. Thermogram of volatile compounds in OFSP under different roasting treatments.

in metabolite profiles across groups. K-means cluster analysis revealed discernible trends in OFSP following treatment under different roasting conditions, classifying all metabolites into eight subclasses accordingly. Notably, the first subclass exhibited the highest number of metabolites (Fig. 4F), with significantly lower median values observed in T180 and T240 treatments, while opposite trends were observed for metabolites within the second and sixth subclasses.

The OPLS-DS model was developed to accurately capture the impact of various roasting conditions on OFSP, with  $\rm R^2X=0.334;\,R^2Y=0.999;$  and  $\rm Q^2=0.603$  as the corresponding parameters. Notably, the OPLS-DA score plots exhibited distinct separation among different sample groups (Fig. 4D), indicating significant variations in OFSP metabolites under diverse roasting conditions, mainly hydrocarbons and ketones.

This finding contradicts the results of previous studies, which



**Fig. 6.** Analysis of differential compounds in OFSP under different roasting treatments. (A) Heat map of differential substances. (B) The number of differential substances. The red color indicates the number of substances with higher relative content compared with the two groups; the blue color indicates the number of substances with lower relative content compared with the two groups. (C) Differential volcano plots. ( $C_1$  is T180vsT210,  $C_2$  is T180vsT240, and  $C_3$  is T210vsT240). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

reported an increase in the content of different compounds in sweet potatoes after roasting (Yao et al., 2023), which may be due to differences in sweet potato varieties as well as roasting temperatures.

Based on the magnitude of VIP values, several metabolites with significant differences were determined: 2-methyl-heptane (2.04), followed by 2,7-dimethyl-octane (1.89), 2,6,11-trimethyl-dodecane (1.84), methacryloyl chloride (1.75), and 6-methyl-6-(5-methylfuran-2-yl)-heptane-2-one (1.71) (Fig. 6A and C). This could be explained that lipid oxidation caused by heat treatment, leading to the formation of shortchain monomers and carbonyl compounds (Hwang, 2015).

In the comparison between T180 and T210, a total of 37 differential compounds were identified, with 17 showing up-regulated expression and 20 showing down-regulated expression. When comparing T180 to T240, we observed 59 differential compounds, among which 26 were up-regulated and 33 were down-regulated. Comparing T210 to T240 revealed a total of 32 differential compounds, with 21 being up-regulated and 11 being down-regulated (Fig. 6B). These findings suggest significant flavor variations between roasted samples of T180 and T240 while indicating that the flavor profile of T180 is more similar to that of T210.

#### 3.4. Carotenoids content

The carotenoid content and composition in OFSP during roasting were qualitatively and quantitatively analyzed using LC-MS/MS analysis. The PCA plot of the carotenoid content revealed that PC1 and PC2 accounted for 45.95 % and 18.69 % of the total variance, respectively (Fig. 2 A2). All samples exhibited distinct separation and clustering within a single class, indicating significant variations in carotenoid content among different sample groups. The total carotenoid content in OFSP decreased in T180M (85.62 µg/g) and exhibited a slight increase in T180 (97.33  $\mu$ g/g), yet remained lower than that of T0 (106.98  $\mu$ g/g). Conversely, the total carotenoid content increased in T210M (108.34  $\mu g/g$ ) and T240M (116.60  $\mu g/g$ ), and the total carotenoid content of T240 (73.52  $\mu g/g)$  was inferior to that of T180 (97.33  $\mu g/g)$  and T210  $(86.07 \,\mu\text{g/g})$ . Irrespective of the roasting method employed, preferential synthesis of carotenoids occurred during the intermediate stage of roasting; nevertheless, prolonged roasting led to degradation and subsequent reduction in levels of carotenoids within roasted OFSP.

According to the molecular structure of carotenoids, they were divided into carotenes and xanthophylls (Milani, Basirnejad, Shahbazi, & Bolhassani, 2017). The levels of carotenes and xanthophylls in roasted OFSP were significantly lower compared to raw samples, with fluctuations during the roasting process (Fig. 2 B<sub>2</sub>). A total of 17 carotenoids were detected in both raw and roasted samples, with 13 compounds identified in all the samples. These include three carotenes:  $\gamma$ -carotene,  $\beta$ -carotene, (E/Z)-phytoene, and ten xanthophylls:  $\beta$ -cryptoxanthin palmitate, zeaxanthin, violaxanthin, neoxanthin, lutein,  $\beta$ -cryptoxanthin,  $\delta$ -apo-beta-carotenal, canthaxanthin, echinenone, zeaxanthin palmitate (Fig. 2 C<sub>2</sub>).

The contents of xanthophylls were very low except for lutein and  $\beta$ -cryptoxanthin, and there was no significant difference between raw and roasted OFSP samples. The lutein content change of each group was roughly the same during roasting, and the longer the roasting time, the more lutein was lost. And T180 had the highest lutein content in roasted OFSP. This is inconsistent with the finding that lutein concentrations in potatoes are not affected by cooking (Blessington et al., 2010), suggesting that there may be specificity in the degradation of lutein in different species during cooking. Previous studies have also found that lutein exhibited a high level of heat resistance during thermal

processing, but the lutein content began to decrease significantly with time at processing temperatures above 180 °C (Chutintrasri & Noomhorm, 2007). At 180 °C, the content of  $\beta$ -cryptoxanthin decreased initially and then increased. However, at 210 °C and 240 °C, the content of  $\beta$ -cryptoxanthin exhibited an opposite trend, and the higher the temperature, the more pronounced the change. The highest content of  $\beta$ -cryptoxanthin was found in roasted sample T180, which was almost the same as that in T0. The zeaxanthin content was the lowest in the raw sample and increased significantly during roasting.

Among the various carotenoids, the levels of  $\gamma\text{-carotene}$  and  $\alpha\text{-carotene}$  exhibited a decrease following roasting, with T180 retaining the highest concentrations. The content of (E/Z)-phytoene demonstrated an increase in both T180 and T210 roasted samples but experienced a significant decline after being subjected to roasting at 240 °C.  $\beta\text{-carotene}$  emerged as the most abundant carotenoid in both raw (79.67 µg/g) and roasted samples (71.15 µg/g, 64.17 µg/g, and 54.48 µg/g in T180, T210, and T240 respectively) (Fig. 2 C<sub>2</sub>). This observation can be attributed to the roasting temperature and duration.

Studies had found that carotenoids are unstable and restricted by a variety of influencing factors, processing methods can affect the release and degradation of carotenoids, and high temperatures and the presence of oxygen can accelerate this reaction (Wang et al., 2024). It was also found that carotenoids differ significantly in their susceptibility to degradation and that enzymatic and non-enzymatic cleavage of carotenoids results in the formation of important aromatic compounds that affect the flavor properties of foods (Meléndez-Martínez, Esquivel, & Rodriguez-Amaya, 2023). Moreover, Zhang et al. found a strong correlation between carotenoids and color parameters, and that changes in  $\beta$ -carotene content influenced color changes in roasted sweet potato meat (Zhang et al., 2023). The observed phenomenon may be attributed to the accelerated isomerization and degradation of carotenoids resulting from the increased roasting temperature. In conclusion, T180 exhibited the highest total carotenoid content after roasting, along with superior retention of various carotenoids.

# 3.5. Bioaccessibility and antioxidant properties of $\beta$ -carotene after in vitro digestion

The  $\beta$ -carotene bioaccessibility and antioxidant properties of raw and roasted OFSP samples showed significant differences (P < 0.05) following simulated gastrointestinal and intestinal fluid digestion. T180 and T210 exhibited high bioaccessibility, indicating enhanced absorption and utilization of  $\beta$ -carotene after roasting (Fig. 1E). Compared to raw OFSP, the antioxidant effect of OFSP improved with a decrease in roasting temperature, with higher scavenging rates of DPPH• and ABTS• free radicals. The highest antioxidant effect was observed at T180. Free radical scavenging activity is commonly expressed as a percentage of scavenged free radicals or as the concentration of antioxidants required to scavenge 50 % of the free radicals (IC50). The IC50 values for DPPH• and ABTS•+ indicated that roasting diminished the antioxidant properties of OFSP, with this effect becoming more pronounced with increasing roasting temperature and time. Similar to its free radical scavenging activity, the FRAP-reducing capacity of OFSP also exhibited significant temperature dependence, with T180 demonstrating the highest reducing capacity (Fig. 1F).

This phenomenon may be attributed to the positive correlation between roasting temperature and isomerization as well as degradation of carotenoids, consequently impacting the antioxidant capacity and bioaccessibility of the sample post-digestion. Heat treatment had been reported to reduce carotenoid content but may help improve carotenoid bioavailability, and the core factors affecting the relative bioavailability of carotenoids are carotenoid intake, composition, etc. (Geng et al., 2023; Maiani et al., 2009). Zhang et al. also found that food microstructure and matrix components affect carotenoid bioaccessibility, and promoted release, solubilization, and micellization of carotenoids may enhance it (Zhang et al., 2024). Therefore, since T180 retained the most contents of  $\beta$ -carotene,  $\beta$ -cryptoxanthin, and lutein on its own, this leads to its optimal antioxidant capacity. The lower temperatures and prolonged roasting conditions facilitated the enhanced absorption and release of carotenoids, thereby augmenting the antioxidant properties and bioaccessibility of digested OFSP.

#### 4. Conclusion

This study evaluated the chemical composition of OFSP during three roasting methods ((180  $^{\circ}\text{C}/70$  min (T180), 210  $^{\circ}\text{C}/50$  min (T210), and 240  $^{\circ}\text{C}/30$  min (T240)), as well as antioxidant activity and flavor changes after roasting. Among all samples, T210 sweet potatoes had the highest content of soluble polysaccharides, especially glucose and fructose. These sugars endow T210 with a moderately sweet taste. In addition, T210 and T180 accumulated a greater variety of volatile compounds and produced better flavors. While T180 demonstrated the most outstanding performance in terms of antioxidant capacity, T210 still hold an edge in this aspect when contrasted with T240. In conclusion, this study found that 210  $^{\circ}\text{C}/50$  min roasting conditions seemed to be the best condition for roasting OFSP, with moderate sweetness and pleasant flavor profile.

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# CRediT authorship contribution statement

Sinian He: Writing – original draft, Methodology, Investigation, Formal analysis, Data curation. Shengsheng He: Writing – review & editing, Methodology. Liya Niu: Writing – review & editing, Methodology. Chao Sun: Writing – review & editing. Zicong Zeng: Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

# 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

Data will be made available on request.

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