



Optimization of processing technology using response surface methodology and physicochemical properties of roasted sweet potato



Feina Hou^{a,b}, Taihua Mu^{a,*}, Mengmei Ma^a, Christophe Blecker^b

^a Institute of Food Science and Technology, Chinese Academy of Agricultural Sciences, Key Laboratory of Agro-Products Processing, Ministry of Agriculture, No. 2 Yuan Ming Yuan West Road, Haidian District, PO Box 5109, Beijing 100193, China

^b Department of Food Science and Formulation, Gembloux Agro-Bio Tech, Université de Liège, Passage des Déportés 2, Gembloux, Belgium

ARTICLE INFO

Chemical compounds studied in this article:

Chlorogenic acid (PubChem CID:1794427)

Sulfuric acid (PubChem CID:1118)

Acetonitrile (PubChem CID:6342)

Disodium hydrogen phosphate (PubChem CID:24203)

Acetic acid (PubChem CID:176)

Sodium hydroxide (PubChem CID:14798)

Hydrochloric acid (PubChem CID:313)

Alpha-D-glucose (PubChem CID:79025)

Sodium carbonate (PubChem CID:10340)

Ethanol (PubChem CID:702)

Keywords:

Response surface methodology

Roasted sweet potato

Nutritional composition

Volatile compounds

Moisture migration

ABSTRACT

The study evaluated the optimal condition for roasting sweet potato using response surface methodology. Proximate composition, antioxidant activity, volatile compounds, and water migration of roasted sweet potato were also determined. The results revealed that the optimal roasting condition included a roasting time of 40 min, a roasting temperature of 235 °C, and a roasting speed of 40 rad/min, the reducing sugar and vitamin C of roasted sweet potato obtained under optimal condition was 47.79 g/100 g (DW) and 60.25 mg/100 g (DW), respectively. After roasting, starch, protein and vitamin C content of sweet potato were significantly decreased, while total phenolic content and antioxidant activity were increased. 2-Methyl butanal was the main aromatic compound. Low-field NMR indicated that the proportion of free water increased and relaxation times (T_2) were decreased after roast process, indicated that the bound water in sweet potato was diffused from inside to outside, thus the texture became softer.

1. Introduction

Sweet potato (*Ipomoea batatas* (L.) Lam) is the fifth food crop worldwide after maize, wheat, rice and potato. In China, the production of sweet potato was 70.79 million tonnes in 2016, which was the largest in the world (FAOSTAT, 2017). Sweet potato is rich in many kinds of nutritional components, such as starch, protein, dietary fiber, phenolics, vitamins, and minerals, etc, but lower fat content. What's more, sweet potato also exhibits some physiological properties, for example, regulating blood glucose and lipid, improving immunity, protecting from cancers and oxidation, etc., thus gaining much more extensive attention and researches in recent years (Wang, Nie, & Zhu, 2016). In addition, the applications of sweet potato have diversified considerably, many scholars have also focused on different drying and cooking methods on the nutritional composition and physicochemical properties of sweet potato. For example, Tang, Cai, and Xu (2015) compared the different thermal treatment, such as steaming, roasting and boiling on the

antioxidant activity of sweet potatoes. Wang and Kays (2001) analyzed the effects of baking, boiling and microwave treatment on volatile compounds of sweet potatoes.

Roasting is the most popular cooking method of sweet potatoes (Collins, Liao, & Penfield, 1995), as a snack food with a long history in China, roasted sweet potato exhibits attractive flavor and cheerful colour which is induced by maillard reaction (Xiao et al., 2014). What's more, the process of roasting could increase the antioxidants content of sweet potato, such as anthocyanins and total phenolics (Blessington et al., 2010). Thus, roasted sweet potato could attract much more consumers than other kinds of sweet potato products.

Different processing conditions during roasting may have great effect on the qualities of roasted products. For example, Shi, Dean, Davis, Sandeep, and Sanders (2018) found that different temperature/time combinations could affect the physicochemical properties of roasted peanuts. Kim, Ko, Kang, and Park (2018) reported that different roasting degree has great influence on volatile flavor substances of

* Corresponding author.

E-mail addresses: mutaihua@126.com, mutaihua@163.com (T. Mu).

<https://doi.org/10.1016/j.foodchem.2018.11.034>

Received 10 July 2018; Received in revised form 2 November 2018; Accepted 6 November 2018

Available online 07 November 2018

0308-8146/ © 2018 Elsevier Ltd. All rights reserved.

coffee, they found that the concentration of dimethyl sulfide slightly decreased in the medium-dark roast, but increased again in the deep-dark roast. However, the optimal roasting condition for sweet potato has not been reported, and the changes of composition, flavor compounds, and moisture transfer of roasted sweet potato have also not been reported. Therefore, it is crucial to evaluate the roasting condition and the above changes during roasting.

Response surface methodology (RSM) is an effective technique which is widely used for optimizing the process parameters. The significant advantage of RSM is that it allows the evaluation of the independent variables and their interactions on the dependent variables with the reduced number of trials (Alara, Abdurahman, & Olalere, 2018). RSM is often used to optimize the processing parameters and has the advantages of fewer experiments and higher accuracy. For example, Mendes, de Menezes, Aparecida, and Da Silva (2001) optimized the roasting condition for chromatic coordinates of robusta coffee by RSM, predictive models were also obtained for the instrumental measurement of the colour of the beans and ground coffee. Raigar, Upadhyay, and Mishra (2017) evaluated the changes of quality characteristics (moisture, hardness and color) of peanuts during the process of microwave roasting, and determined the optimal roasting condition of peanuts by RSM.

In this study, RSM was used to study the effects of different roasting parameters, such as roasting temperature, roasting time and roasting speed on reducing sugar and vitamin C content of roasted sweet potatoes, and determine the optimal technological parameters of roasted sweet potatoes. On this basis, proximate compositions (protein, fat, ash, total starch, total dietary fiber, reducing sugar and vitamin C), total phenolic content (TPC), antioxidant activity and weight-loss ratio of roasted sweet potato were determined under the optimal process. In addition, GC-MS and low-field NMR was used to determine the volatile compounds and moisture migration of roasted sweet potato.

2. Materials and methods

2.1. Materials and reagents

The fresh sweet potato samples (Yanshu No.25, with a flesh color of orange) were provided by Hebei Academy of Agriculture and Forestry Sciences (Shijiazhuang, Hebei Province, China) and were collected from Shandong province, the harvest time was November. The fresh sweet potato roots were selected according to the weight which was about 150 g, washed with clean water and placed in a roaster (model No.128, Shengzhou machinery and equipment, Co., Ltd, Shenyang, China) to obtain roasted sweet potatoes. After cooling to room temperature, the roasted sweet potatoes were freeze-dried (SIM-FD5, the Siemon Company, Los Angeles, America), and then were smashed and screened through 100 mesh sieve for further analysis.

The trolox (> 98%), chlorogenic acid (≥95%), and ascorbic acid standard (≥98.9%) were HPLC grade and were purchased from Sigma-Aldrich (St. Louis MO, USA & Shanghai, China). All other reagents were of analytical grade.

2.2. Optimization of the roasting parameters

The effects of roasting time, temperature, and speed on the reducing sugar, ash, vitamin C, fat, protein, weight-loss ratio, total phenolic content (TPC), and antioxidant activity were determined by single factor test, the content of reducing sugar and vitamin C was chosen as the response value for RSM on the basis of main component analysis (the results were not shown). A Box-Behnken design with three independent factors (X_1 , roasting time; X_2 , roasting temperature; X_3 , roasting speed) at three variation levels was performed. The correspondence between the coded and uncoded values was obtained by Eq. (1):

$$X_i = (X_i - X_0) / \Delta X_i \quad (1)$$

where x_i is the coded value of the variable, X_i is the actual value of the variable, X_0 is the actual value of X_i at the central point and ΔX_i as the value of the change.

The range of roasting time (X_1), roasting temperature (X_2), and roasting speed (X_3) was used to prepare the 17 experiments which including 12 factorial points (levels ± 1) and 5 replicates of the central point. The reducing sugar (Y_1) and vitamin C (Y_2) were response value. The design expert (version 8.0) software were used to analyze the experimental data (www.statease.com). Experimental data were fitted to a second-order polynomial model and regression coefficients obtained. The generalized second-order polynomial model used in the response surface analysis was as follows [Eq. (2)]:

$$Y = \beta_0 + \sum_{i=1}^3 \beta_i X_i + \sum_{i=1}^3 \beta_{ii} X_i^2 + \sum_{i < j=1}^3 \beta_{ij} X_i X_j \quad (2)$$

where Y is the response value, X_i is the independent variable, and β_0 , β_i , β_{ii} , β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively. The design expert software was used to generate response surfaces and contour plots while holding a variable constant in the second-order polynomial model. When the results showed a saddle point in response surfaces the ridge analysis of design expert RSREG procedure was used to compute the estimated ridge of the optimum response (Liyana-Pathirana & Shahidi, 2005).

2.3 Weight-loss ratio

The weight of sweet potatoes were weighed and recorded before and after roasting process. The weight-loss ratio (%) was calculated according to the following Eq.(3):

$$\text{Weight-loss ratio (\%)} = (W_1 - W_2) \times 100\% / W_1 \quad (3)$$

where W_1 and W_2 was sweet potato weight before and after roasting process, respectively.

2.4. Nutritional composition

The content of ash, fat, protein, total dietary fiber, reducing sugar, and vitamin C were determined according to the AOAC methods (Association of Official Analytical Chemists, 2000). Ash content was determined by weighing samples before and after heat treatment at 550 °C in a muffle furnace for 6 h (AOAC method 923.03). Fat content was determined by soxhlet extraction method according to AOAC method 960.39. Protein was determined according to AOAC method 976.05 assessed by the micro-Kjeldahl method, with nitrogen to protein conversion factor of 6.25. Total dietary fiber (TDF) was determined by digesting the sample with α -amylase (AOAC method 991.43). Reducing sugar content was determined by AOAC method 945.66. Vitamin C (total ascorbic acid) was determined according to the AOAC method 984.26.

2.5. TPC

TPC was measured by the Folin-Ciocalteu method (Sun, Mu, Xi, Zhang, & Chen, 2014). Briefly, 0.1 g lyophilized powders was mixed with 2.0 mL of 70% (v/v) ethanol, and extracted with ultrasonic machine for 30 min at 50 °C. After centrifugation at 5000g for 10 min at 4 °C, the residue was re-extracted for another 2 times with 70% ethanol as mentioned above. Three extracts were pooled and diluted 10 times with distilled water. An aliquot (0.5 mL) extracts was mixed with 1.0 mL of Folin-Ciocalteu reagent and diluted 10 times with distilled water, the mixtures were subsequently allowed to react for 30 min at 30 °C. Then, 2.0 mL of Na_2CO_3 (10%, w/v) was added into the mixtures and continued to react for 30 min. After that, the absorbance was detected at 736 nm using a UV1101 spectrophotometer (Hitachi, Japan).

A calibration curve consisting of chlorogenic acid (CHA) standards (concentrations ranging from 0.02 to 0.10 mg/mL) was prepared. TPC was expressed as grams of CHA equivalents (CHAE) on a DW basis (g CHAE/100 g).

2.6. Antioxidant capacity

Antioxidant capacity was determined by oxygen radical absorbance capacity (ORAC) assay and was carried out following the procedure established by Prior et al. (2003) with slight modifications. All reagents and samples in this experiment were dissolved and diluted with phosphate buffer (0.075 M, pH7.4). Briefly, 20 μ L sample solutions (extraction process was the same as TPC) were mixed with 20 μ L phosphate buffer and then was added 20 μ L 63 nmol/L sodium fluorescein solution in a clear 96-well microplate and incubated at 37 °C. After reaction for 15 min, 140 μ L 18.28 mmol/L AAPH solution was rapidly added to the well and mixed well. Then, the microplate was placed in the multi-functional microplate reader (Hidex Ltd. Co., Finland).

The system was set in fluorescence mode, and the fluorescence intensity of each well was read 60 times at 2 min intervals. The emission and excitation filter wavelengths were set at 535 nm and 485 nm and the detection temperature was 37 °C.

The antioxidant capacity was calculated according to the Eqs. (4)–(6), and was expressed as μ g trolox equivalent of sample (trolox equivalent, TE) per mg (μ g TE/mg).

$$F_i = f_i(+AAPH)/f_i(-AAPH) \quad (4)$$

$$AUC = 2 \times (F_0 + F_1 + \dots + F_n) - F_1 - F_n \quad (5)$$

$$\text{netAUC} = AUC_{\text{sample}} - AUC_{\text{blank}} \quad (6)$$

where f_i is the fluorescence intensity of the reaction solution; F_i is the relative fluorescence intensity of the reaction solution; AUC is the area under the curve; in this study, the interval time and the value was 2; and netAUC is the net area under the curve between the blank and the samples.

2.7. Nuclear magnetic resonance

The water status of the fresh and roasted sweet potato samples were determined by nuclear magnetic resonance (NMR). NMR relaxation measurements were carried out following the procedure established by Xu, Zhang, Bhandari, Cheng, and Sun (2015) using a low-field NMR analyser MesoMR (Niumag Electric Corporation, Shanghai, China). The transverse relaxation time (T_2) was determined using the Carr-Purcell-Meiboom-Gill pulse sequence. Optimal pulse parameters were as follows: P1 = 18 μ s, SF = 23 MHz, SW = 250 kHz, TD = 459,942, DR = 1, DRG1 = 3, TW = 3500,000 ms, NS = 4, NECH = 8000. Each measurement was performed in triplicate.

2.8. GC–MS analysis

Volatile compounds were identified by GC–MS system (GCMS-QP2010 Plus, Shimadzu, Japan) using a solid phase micro extraction isolation technique. One gram of sample was heated to 60 °C in a vial and the headspace was sampled with a 65 μ m polydimethylsiloxane SPME fiber (Supelco, Bellefonte, PA) for 40 min. The injection temperature was 200 °C, the oven temperature was at 40 °C held for 1 min, increased to 160 °C at a rate of 6 °C/min and then to 250 °C at a rate of 10 °C/min. The scan mode was used to detect all the compounds in the range m/z 35–500 atomic mass unit. The identification of volatile compounds was verified by comparison of the mass spectral data obtained with those in the NIST library (Ver. 11, Ringoes, NJ, USA). The contents of the volatile compounds were expressed as relative peak areas.

2.9. Statistical analysis

Each test was carried out in triplicate. The data were expressed as the mean \pm standard deviation (SD), and one-way analysis of variance was conducted by Duncan's multiple range test using SAS 9.2 software (SAS Institute Inc., Cary, NC, USA). The means were considered to be significantly different at $p < 0.05$.

3. Results and discussion

3.1. Principal component analysis

The 8 quality characteristics (including weight-loss ratio, protein, fat, ash, reducing sugar, vitamin C, total phenols content and antioxidant activity) were consolidated into 3 principal components which accounted for 98.56% of total variation. Principal component 1(PC1) accounted for 72.42% of the variance, whereas principal component 2(PC2) accounted for the remaining 15.55% of the variance in the data matrix, indicating that the difference in various samples was mainly reflected by PC1 and PC2.

The contribution of the first principal component is vitamin C (0.8607), followed by reducing sugar (–0.4767), which can be thought of vitamin C and reducing sugar is the representation evaluation index of the PC1. The contribution of the second principal component is reducing sugar (0.8607), followed by vitamin C (0.4767), which can be thought of reducing sugar and vitamin C is the representation evaluation index of the PC2. In conclusion, the quality evaluation indexes of roast sweet potato were reducing sugar content and vitamin C content.

3.2. Accuracy and variance analysis of the regression model

3.2.1. The content of reducing sugar of roasted sweet potato

The content of reducing sugar (Y_1) was shown in Table 1, and the analysis of variance (ANOVA) of the result of Box-Behnken was summarized in Table 2. The fitted quadratic model for reducing sugar (Y_1) was estimated by RSM, taking into account only the significant terms, which was shown in Eq. (7):

$$Y_1 (\text{reducing sugar, g/100g DW}) = 367.34 + 1.01X_2 + 1.92X_3 - 0.0081X_1X_2 - 0.025X_1X_3 - 0.0036X_2X_3 - 0.11X_1^2 - 0.00089X_2^2 \quad (7)$$

The quadratic regression model for reducing sugar resulted in a determination coefficient ($R^2 = 0.9875$), indicating that 98.75% of the variation could be explained excellently (Wai, Alkarkhi, & Easa, 2010). The lack of fit associated with P-values of 0.64, demonstrated a non-significance, supporting that the model fits with the data. A P-value lower than 0.0001 was found, demonstrating again the high significance of the regression model and can be used to optimize the variables. More importantly, roasting time (X_1) and roasting speed (X_3) significantly affected the content of reducing sugar of roasted sweet potatoes, followed by roasting temperature (X_2). The quadratic term (X_1^2 , and X_2^2) was highly significant ($p < 0.01$), and X_1X_2 , X_1X_3 , and X_2X_3 terms was also highly significant ($p < 0.01$). The other terms were insignificant.

3.2.2. The content of vitamin C of roasted sweet potato

The content of vitamin C (Y_2) was also shown in Table 1, and the analysis of variance (ANOVA) of the result of Box-Behnken was summarized in Table 2. The fitted quadratic model for vitamin C (Y_2) was estimated by RSM, neglecting the insignificant terms, which was shown in Eq. (8):

$$Y_2 (\text{vitamin C, mg/100 g DW}) = 161.26 - 3.73X_1 - 0.029X_2 \quad (8)$$

The quadratic regression model for vitamin C resulted in a determination coefficient ($R^2 = 0.9523$), indicating that 95.23% of the

Table 1
Design approach and experimental results of response surface methodology.

Run	Independent variables			Reducing sugar (g/100 g, DW)		Vitamin C (mg/100 g, DW)	
	X ₁ (min)	X ₂ (°C)	X ₃ (rad/min)	Measured	Predicted	Measured	Predicted
1	40	240	50	43.07 ± 0.37	43.74	61.57 ± 0.23	61.94
2	40	200	70	39.00 ± 0.12	39.31	67.52 ± 0.15	65.71
3	45	280	50	40.66 ± 0.45	40.97	52.99 ± 0.08	52.51
4	40	280	30	50.00 ± 0.26	49.69	52.62 ± 0.31	54.43
5	40	280	70	40.21 ± 0.33	39.85	54.61 ± 0.47	53.70
6	45	240	70	35.61 ± 0.24	35.66	55.19 ± 0.39	56.58
7	40	240	50	43.58 ± 0.38	43.74	61.89 ± 0.22	61.94
8	40	240	50	43.10 ± 0.11	43.74	60.58 ± 0.19	61.94
9	40	240	50	44.38 ± 0.41	43.74	63.85 ± 0.12	61.94
10	40	240	50	44.55 ± 0.29	43.74	61.83 ± 0.36	61.94
11	40	200	30	37.27 ± 0.16	37.63	66.13 ± 0.23	67.04
12	35	200	50	35.33 ± 0.35	35.02	72.09 ± 0.19	72.57
13	45	240	30	44.67 ± 0.24	44.68	60.66 ± 0.24	59.33
14	45	200	50	38.26 ± 0.09	37.90	63.66 ± 0.36	64.08
15	35	240	30	40.13 ± 0.34	40.08	66.74 ± 0.11	65.35
16	35	240	70	40.96 ± 0.21	40.96	64.74 ± 0.28	66.06
17	35	280	50	44.19 ± 0.37	44.55	59.94 ± 0.33	59.52

variation can be explained excellently, only 4.77% of the total variation could not be explained (Wai et al., 2010). The lack of fit associated with P-values of 0.1126, demonstrated a non-significance, supporting that the model fits with the data. A P-value was 0.0008, demonstrating again the high significance of the regression model. In the selected levels, roasting temperature (X₂) and roasting time (X₁) significantly affected the content of vitamin C of roasted sweet potatoes, followed by roasting speed (X₃). The quadratic term (X₁², X₂² and X₂₃²) was insignificant (p > 0.05), and X₁X₂, X₁X₃, and X₂X₃ terms was also insignificant (p > 0.05).

3.3. The effects of roasting parameters on the content of reducing sugar and vitamin C

3.3.1. Effects of roasting parameters on reducing sugar content

Three-dimensional response surface plots were chosen to illustrate a visual interaction between independent and dependent variables (Dahmoune et al., 2014). The reducing sugar content of roasted sweet potato ranged from 35.33 to 50.00 g/100 g, depended on the roasting temperature, roasting time and roasting speed and their interaction (Fig. 1a–c).

The plot depicting the interaction influence of the two factors, roasting temperature and roasting time on the reducing sugar content,

was shown in Fig. 1a. It can be seen that the content of reducing sugar increased when roasting temperature was increased. The linear factor of roasting time did not exert a significant effect on reducing sugar content (p-value = 0.46), but its interaction with roasting temperature allowed significant influence on reducing sugar content of roasted sweet potato, with a p-value of 0.0015. In other words, roasting temperature increased the content of reducing sugar with a middle roasting time (about 39 min). Roasting temperature was the most important factor (p-value < 0.0001) which could affect the content of reducing sugar efficiently. With increase of temperature, the sucrose was decomposed into glucose and fructose after dehydration. On the other hand, the degree of Maillard reaction and the degradation of starch was increased, thus leading to the formation of maltose (Cárnara, Diez, & Torija, 1995).

Fig. 1b shows the interaction effect of roasting time and speed on the reducing sugar content of roasted sweet potato. The result indicated that the reducing sugar content changed little with the increase of roasting speed when the roasting time is short. However, the reducing sugar content decreased significantly with the increase of roasting time. What's more, roasting speed is also a factor which affecting the reducing sugar content, which might be because it could influence the uniformity of temperature.

From Fig. 1c, we can see that reducing sugar content increased with

Table 2
Analysis of variance (ANOVA) for response surface model.

Source	Reducing sugar					Vitamin C				
	Sum of Squares	Degree of freedom	Mean Square	F Value	p-value Prob > F	Sum of Squares	Degree of freedom	Mean Square	F Value	p-value Prob > F
Model	225.55	9	25.06	61.41	< 0.0001	438.72	9	48.75	15.52	0.0008
X ₁	0.25	1	0.25	0.61	0.46	120.20	1	120.20	38.26	0.0005
X ₂	79.38	1	79.38	194.51	< 0.0001	303.07	1	303.07	96.47	< 0.0001
X ₃	33.17	1	33.17	81.28	< 0.0001	2.09	1	2.09	0.67	0.44
X ₁ X ₂	10.43	1	10.43	25.57	0.0015	0.55	1	0.55	0.17	0.69
X ₁ X ₃	24.45	1	24.45	59.92	0.0001	3.01	1	3.01	0.96	0.36
X ₂ X ₃	33.18	1	33.18	81.30	< 0.0001	0.09	1	0.09	0.029	0.87
X ₁ ²	30.73	1	30.73	75.31	< 0.0001	3.56	1	3.56	1.13	0.32
X ₂ ²	8.54	1	8.54	20.93	0.0026	2.02	1	2.02	0.64	0.45
X ₃ ²	2.01	1	2.01	4.94	0.062	4.47	1	4.47	1.42	0.27
Residual	2.86	7	0.41			21.99	7	3.14		
Lack of Fit	0.91	3	0.30	0.62	0.64	16.34	3	5.45	3.86	0.11
Pure Error	1.95	4	0.49							
Cor Total	228.41	16								
R-Squared	0.9875					0.9523				

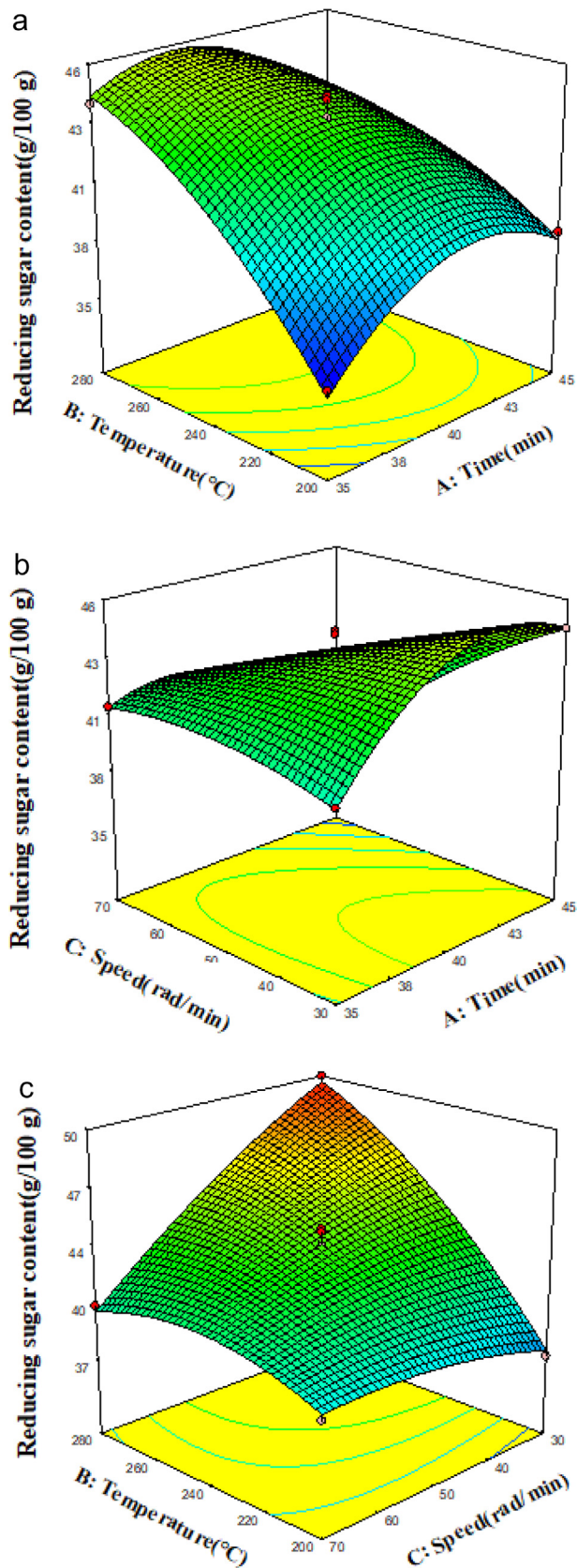


Fig. 1. Response surface for the effect of independent variables on reducing sugar content.

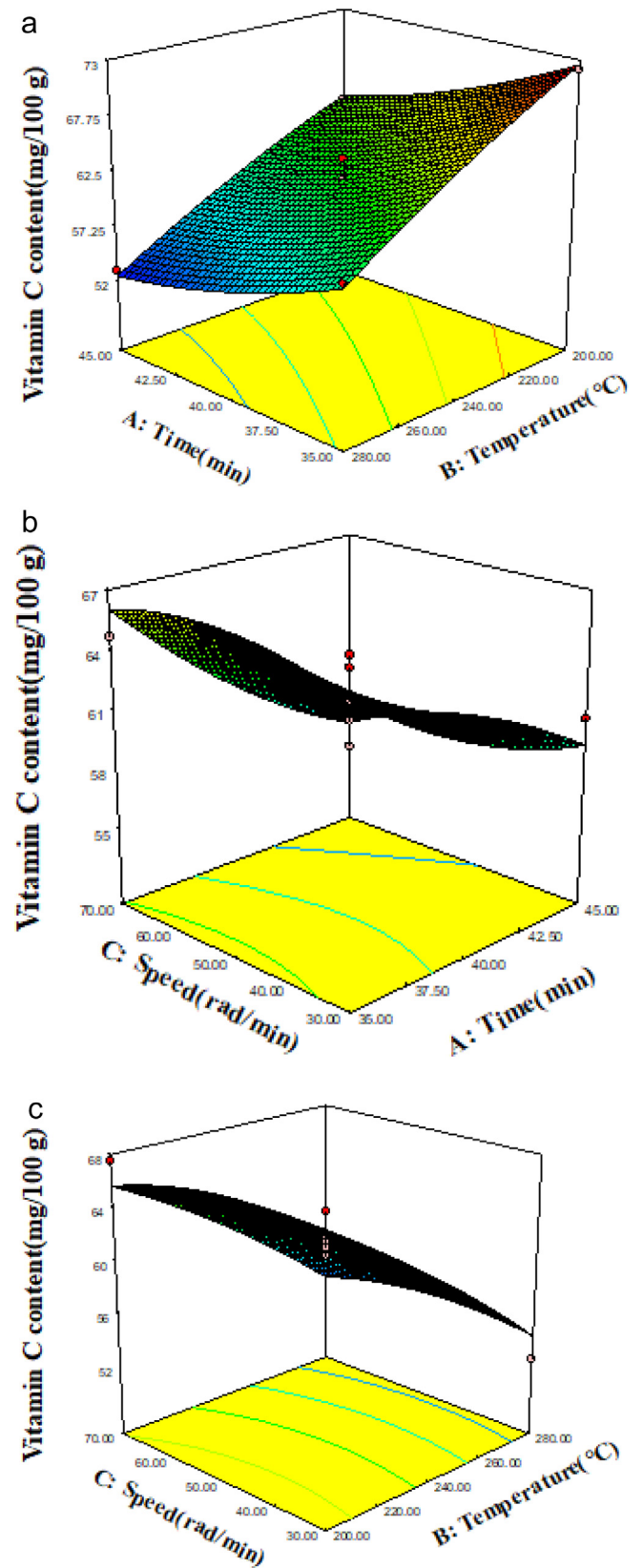


Fig. 2. Response surface for the effect of independent variables on vitamin C content.

the increase of roasting temperature and decrease of roasting speed as roasting time was kept constant at 40 min. Roasting time was another vital factor for the content of reducing sugar. Garza, Ibarz, Pagan, and Giner (1999) reported that the hydroxymethylfurfural (HMF) content increased with treatment time, which was associated with the non-enzymatic browning reaction occurring during the study.

3.3.2. Effects of roasting parameters on vitamin C content

Like reducing sugar, roasting parameters depicting the highest influence on the content of vitamin C were the roasting temperature. In Fig. 2a–c, it can be concluded that although the effect of roasting temperature and roasting time on vitamin C content was obvious (p -value < 0.0001), its interaction was not obvious. The interaction between roasting parameters exerted a little influence on vitamin C content. In addition, the effect of roasting speed on vitamin C content was not significant (p -value = 0.4415).

The plot depicting the influence of the two factors, roasting temperature and roasting time on the vitamin C content was shown in Fig. 2a. As the roasting temperature and time increased, the vitamin C content decreased. From Fig. 2b and c, we can see that roasting speed has little effect on vitamin C content, while the roasting temperature and time increased, the vitamin C content decreased.

Loss of vitamin C occurs primarily by chemical degradation that involves oxidation of ascorbic acid to dehydroascorbic acid (DHAA), followed by hydrolysis to 2,3-diketogulonic acid and further polymerization to form other nutritionally inactive products (Gregory, 1996). Previous study showed that the oxidation process of ascorbic acid could be accelerated during thermal treatment, thus reducing the vitamin C content in fruits and vegetables (Dewanto, Wu, Adom, & Liu, 2002). Therefore, the decrease of vitamin C with increased roasting time and temperature might be attributed to the heating process.

3.4. Verification of predictive models

The optimum value of reducing sugar was 49.73 g/100 g DW, for a roasting temperature of 280 °C, roasting time of 40.63 min and roasting speed of 30 rad/min. For vitamin C, the maximum content predicted by the model was 72.58 mg/100 g DW with respective optimum parameters of 200 °C, 35 min and roasting speed of 52.09 rad/min.

Taking into account processing costs, responses for both reducing sugar and vitamin C were simultaneously optimized by the desirability function. In order to find the optimum parameters that would result in maximum reducing sugar and vitamin C, the desirability profile was carried out. The results indicated that the maximum overall both of reducing sugar and vitamin C could be achieved with a roasting temperature of 235 °C, roasting time of 40 min and roasting speed of 40 rad/min, the maximum content of reducing sugar and vitamin C were 44.03 g/100 g DW, and 62.68 mg/100 g DW, respectively.

In order to validate the reasonability of the model equations, an experiment was carried out in triplicate under the optimal condition. The content of reducing sugar and vitamin C was 47.79 ± 0.43 g/100 g DW and 60.25 ± 0.67 mg/100 g DW, respectively, which was in good agreement with the predicted data.

3.5. Proximate composition and weight-loss ratio

After roasting, fat, reducing sugar, dietary fiber and TPC content were increased significantly (with different letters, $p < 0.05$), starch, protein and vitamin C content were decreased significantly, while ash content and antioxidant activity didn't show significant changes (with the same letter, $p > 0.05$).

The weight-loss ratio of roasted sweet potato was 21.38%, this might be due to the loss of water through evaporation during roasting. In the carbohydrate, starch is the most abundant component, which content was 61.46 g/100 g DW in fresh sweet potato. After roasting, the starch content decreased to 41.65 g/100 g DW, this might be because

the starch was degraded into sugars during the thermal process (Shamla & Nisha, 2017). Compared to fresh sweet potato (9.23 g/100 g DW), the protein content in roasted sweet potato (6.87 g/100 g DW) was also decreased, which could be caused by individual differences of sweet potatoes. What's more, it also might be associated with the involvement of proteins in non-enzymatic browning reaction during the thermal process (Wang et al., 2016). Consequently, as a result of roasting, an improvement in lipid extractability (from 0.63 to 1.08 g/100 g DW) was observed, this might be due to the increased extraction efficiency of coalescent lipids during Soxhlet analysis (Corrales et al., 2017). The ash and dietary fiber content of roasted sweet potato was 3.64 and 8.58 g/100 g DW, respectively.

TPC of fresh sweet potato was 3.34 mg CHAE/g DW, after roasting, the TPC was increased significantly (5.42 mg CHAE/g DW). This could be due to softening or disruption of plant cell walls and the destruction of complex phenolics during thermal treatment. In addition, heat treatment could also cause the complex components breakdown in sweet potato, some individual phenolic substances were released, which could react with Folin-Ciocalteu reagent (Turkmen, Sari, & Velioğlu, 2005). The antioxidant activity of fresh sweet potato was 2.13 μ g TE/mg DW, it was increased slightly after roasting (although there was no statistically significant difference), which was 2.25 μ g TE/mg DW, this might be due to the increase of TPC, and some other antioxidant components generated during the maillard reaction (Smith & Alfawaz, 1995).

Compared to the vitamin C content of fresh sweet potato (83.73 mg/100 g DW), After roasting, it reduced to 60.25 mg/100 g DW. Thermal processing resulted in loss of vitamin C content, as it mentioned before. Reducing sugar content of fresh and roasted sweet potato was 11.09 and 47.79 g/100 g DW, respectively. Results indicated that sugar content increased significantly caused by the degradation of starch and formation of maltose (Chan et al., 2014).

3.6. GC–MS analysis

The volatile compounds and their relative peak areas of roasted sweet potato were determined by GC–MS, the result indicated that thirty-eight volatile compounds of roasted sweet potato were detected.

One of the main volatile compounds was aldehydes. Branched aldehydes probably originate from amino acid degradation, such as non-enzymic processes (Griffith & Hammond, 1989). 2-Methyl butanal (36.04%), produced from isoleucine, which were responsible for the complex and harsh flavors, which were reported by Dunn and Lindsay (1985) previously. Benzaldehyde (7.24%) and benzeneacetaldehyde (12.76%) contributed to the sweet and flowery flavors of roasted sweet potato. Mo, Xu, and Fan (2010) considered that benzaldehyde came from microbial catabolism of amino acids, and benzeneacetaldehyde was an enzymolysis product of phenylalanine. In addition, the previous report also showed that the content of benzaldehyde and benzeneacetaldehyde in malt increased greatly during the roasting stage, and also increased with the increasing roasting time (Dong et al., 2013).

Furfural (3.57%), 2-furanmethanol (5.15%) and 5-methyl-2-furanmethanol (0.40%) were the products of maillard reaction, which contributed to a sweet and caramelized odor (Nakamura, Ono, Yagi, & Miyazawa, 2013). Maltol was demonstrated previously to be an important in the aroma of baked sweet potato (Wang & Kays, 2000), however, this compound was not detected in this study. Our results were similar with Nakamura et al. (2013), who studied the volatile compounds in boiled sweet potato obtained from 3 cultivars, which was Ayamurasaki, Beniazuma and Simon 1, respectively, and found that maltol was detected only in one species (Ayamurasaki: 2.1%). According to Sun, Severson, Schlotzhauer, and Kays (1995), maltol was not a volatile thermolytic product from the maltose and other sugar, which required the presence of an appropriate nitrogen source, such as amino acids or proteins. This might be caused by differences in the sweet potato cultivars, and the process of roasted and pretreatment. It

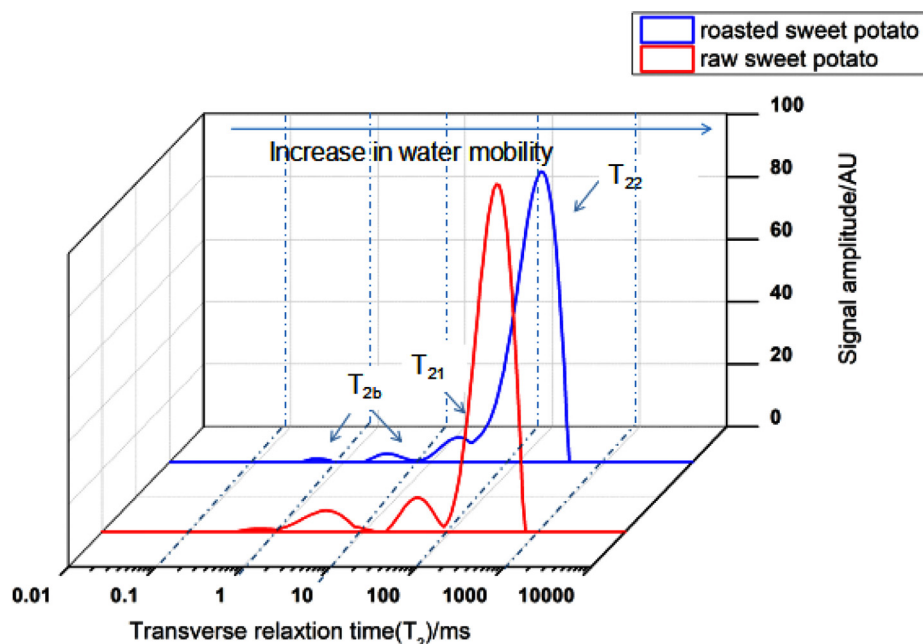


Fig. 3. Typical distribution of T_2 relaxation of fresh and roasted sweet potato.

also might be related to the measuring instruments and procedures.

3.7. Low-field NMR analysis

The distribution and mobility of water molecules of sweet potato before and after roasting was determined by low-field NMR, and the result was shown in Fig. 3 and Table 3. In this study, 3 distinct regions were observed, which presented 3 different water distribution states (Fig. 3). T_{2b} expressed the bound water which was relevant to the water contained in cell walls, and it was tightly bound by strong H-bonds. T_{21} mainly corresponded to immobilized water which was associated with the water contained in cytoplasm and extra-cellular, which was strongly bound to the monolayer. T_{22} stranded for the free water, which expressed the water contained in the vacuole, which was weakly bound to the product (Wang, Xu, Wei, & Zeng, 2018).

In Fig. 3, the curve of the roasted sweet potato shifted toward the left to X-axis compared to the fresh sample, which meant T_2 relaxation time of sweet potato sample decrease. After roasting, this changes also could be seen in Table 3. This was probably due to the water runoff during roasting process. The moisture distribution and composition was also shown in Table 3 (S_{2b} , S_{21} , S_{22}). Compared to fresh sample, it is clearly noted that the proportion of free water in roasted sweet potato increased significantly, and accounted for approximately 95% of the total water, while the proportion of bound water and immobilized water of roasted sample decreased significantly. This might be due to that immobile water changed into free water constantly during roasting, the internal free water gradient was higher than that of outside, which promoted the internal water diffusion to the outside and loss through evaporation. In addition, the destruction of cell walls by

thermal process would lead to the chemical exchange between hydroxyl protons and water on the cell wall polysaccharides (hemicellulose, cellulose and pectin) and water loss from the cell walls (Wang et al., 2018).

4. Conclusion

In this work, the influences of roasting factors on the reducing sugar and vitamin C content were studied using response surface methodology. The obtained results showed that roasting time, roasting temperature and roasting speed affected the measured responses, and the optimal roasting conditions included a roasting time of 40 min, a roasting temperature of 235 °C, and a roasting speed of 40 rad/min. Under the optimal conditions, the reducing sugar of 47.79 ± 0.43 g/100 g DW, vitamin C content of 60.25 ± 0.67 mg/100 g DW. In addition, 38 volatile compounds were identified in roasted sweet potato by GC-MS, 2-methyl butanal was the main aromatic compound. Low-field NMR results showed that roasting changes the water distribution, the proportion of free water increased and relaxation times (T_2) were decreased after roasting process.

Acknowledgement

The authors gratefully acknowledge the earmarked fund for China Agriculture Research System (CARS-10-B21) and the National Key R&D Program of China (2016YFE0133600). The authors are grateful for the support provided by the Institute of Food Science and Technology, Chinese Academy of Agricultural Sciences.

Table 3

The relaxation times and relative areas of T_{2b} , T_{21} and T_{22} populations of raw and roasted sweet potato.

Mode	T_2 (ms)			Area of T_2 (%)		
	T_{2b}	T_{21}	T_{22}	S_{2b}	S_{21}	S_{22}
Raw sweet potato	3.42 ± 0.33^a	36.47 ± 1.46^a	311.54 ± 16.40^a	6.47 ± 0.41^a	6.66 ± 0.31^a	86.87 ± 0.72^b
Roasted sweet potato	3.29 ± 0.16^a	20.03 ± 0.98^b	172.36 ± 8.46^b	1.60 ± 0.41^b	3.35 ± 1.99^b	95.04 ± 2.40^a

Data are means \pm standard deviation (SD) ($n \geq 3$).

Values (a and b) within same column with different letters are significantly different ($p < 0.05$).

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.foodchem.2018.11.034>.

References

- Alara, O. R., Abdurahman, N. H., & Olalere, O. A. (2018). Optimization of microwave-assisted extraction of flavonoids and antioxidants from *Vernonia amygdalina* leaf using response surface methodology. *Food and Bioprocess Technology*, *107*, 36–48.
- AOAC (Association of Analytical Chemists) (2000). *Official methods of analysis* (17th ed.). Gaithersburg, MD, USA: AOAC International.
- Blessington, T., Nzaramba, M. N., Scheuring, D. C., Hale, A. L., Reddivari, L., & Miller, J. C. (2010). Cooking methods and storage treatments of potato: Effects on carotenoids, antioxidant activity, and phenolics. *American Journal of Potato Research*, *87*(6), 479–491.
- Cárnara, M., Diez, C., & Torija, E. (1995). Chemical characterization of pineapple juices and nectars. Principal components analysis. *Food Chemistry*, *54*(1), 93–100.
- Chan, C. F., Chiang, C. M., Lai, Y. C., Huang, C. L., Kao, S. C., & Liao, W. C. (2014). Changes in sugar composition during baking and their effects on sensory attributes of baked sweet potatoes. *Journal of Food Science and Technology*, *51*(12), 4072–4077.
- Collins, J. L., Liao, J. Y., & Penfield, M. P. (1995). Chemical, physical and sensory attributes of formed and frozen, baked sweetpotato. *Journal of Food Science*, *60*(3), 465–467.
- Corrales, C. V., Lebrun, M., Vaillant, F., Madec, M. N., Lortal, S., Pérez, A. M., & Fliedel, G. (2017). Key odor and physicochemical characteristics of raw and roasted jicaro seeds (*Crescentia alata* KHB). *Food Research International*, *96*, 113–120.
- Dahmoune, F., Spigno, G., Moussi, K., Remini, H., Cherbal, A., & Madani, K. (2014). Pistacia lentiscus leaves as a source of phenolic compounds: Microwave-assisted extraction optimized and compared with ultrasound-assisted and conventional solvent extraction. *Industrial Crops and Products*, *61*, 31–40.
- Dewanto, V., Wu, X., Adom, K. K., & Liu, R. H. (2002). Thermal processing enhances the nutritional value of tomatoes by increasing total antioxidant activity. *Journal of Agricultural and Food Chemistry*, *50*(10), 3010–3014.
- Dong, L., Piao, Y., Zhang, X., Zhao, C., Hou, Y., & Shi, Z. (2013). Analysis of volatile compounds from a malting process using headspace solid-phase micro-extraction and GC–MS. *Food Research International*, *51*(2), 783–789.
- Dunn, H. C., & Lindsay, R. C. (1985). Evaluation of the role of microbial Strecker-derived aroma compounds in unclean-type flavors of cheddar cheese. *Journal of Dairy Science*, *68*(11), 2859–2874.
- FAO (Food and Agriculture Organization) (2017). *Food and agricultural commodities production*. Food and Agriculture Organization of the United Nations.
- Garza, S., Ibarz, A., Pagan, J., & Giner, J. (1999). Non-enzymatic browning in peach puree during heating. *Food Research International*, *32*(5), 335–343.
- Gregory, J. F. (1996). Vitamins. In O. R. Fennema (Ed.). *Food Chemistry* (pp. 531–616). (3rd ed.). New York: Dekker.
- Griffith, R., & Hammond, E. G. (1989). Generation of swiss cheese flavor components by the reaction of amino acids with carbonyl compounds 1. *Journal of Dairy Science*, *72*(3), 604–613.
- Kim, S. Y., Ko, J. A., Kang, B. S., & Park, H. J. (2018). Prediction of key aroma development in coffees roasted to different degrees by colorimetric sensor array. *Food Chemistry*, *240*, 808–816.
- Liyana-Pathirana, C., & Shahidi, F. (2005). Optimization of extraction of phenolic compounds from wheat using response surface methodology. *Food Chemistry*, *93*(1), 47–56.
- Mendes, L. C., de Menezes, H. C., Aparecida, M., & Da Silva, A. P. (2001). Optimization of the roasting of robusta coffee (*C. canephora conillon*) using acceptability tests and RSM. *Food Quality and Preference*, *12*(2), 153–162.
- Mo, X., Xu, Y., & Fan, W. (2010). Characterization of aroma compounds in Chinese rice wine Qu by solvent-assisted flavor evaporation and headspace solid-phase micro-extraction. *Journal of Agricultural and Food Chemistry*, *58*(4), 2462–2469.
- Nakamura, A., Ono, T., Yagi, N., & Miyazawa, M. (2013). Volatile compounds with characteristic aroma of boiled sweet potato. *Journal of Essential Oil Research*, *25*(6), 497–505.
- Prior, R. L., Hoang, H. A., Gu, L., Wu, X., Bacchiocca, M., Howard, L., & Jacob, R. (2003). Assays for hydrophilic and lipophilic antioxidant capacity (oxygen radical absorbance capacity (ORACFL)) of plasma and other biological and food samples. *Journal of Agricultural and Food Chemistry*, *51*(11), 3273–3279.
- Raigar, R. K., Upadhyay, R., & Mishra, H. N. (2017). Optimization of microwave roasting of peanuts and evaluation of its physicochemical and sensory attributes. *Journal of Food Science and Technology*, *54*(7), 2145–2155.
- Shamla, L., & Nisha, P. (2017). Acrylamide formation in plantain (*Musa paradisiaca*) chips influenced by different ripening stages: A correlation study with respect to reducing sugars, amino acids and phenolic content. *Food Chemistry*, *222*, 53–60.
- Shi, X., Dean, L. O., Davis, J. P., Sandeep, K. P., & Sanders, T. H. (2018). The effects of different dry roast parameters on peanut quality using an industrial belt-type roaster simulator. *Food Chemistry*, *240*, 974–979.
- Smith, J. S., & Alfawaz, M. (1995). Antioxidative activity of Maillard reaction products in cooked ground beef, sensory and TBA values. *Journal of Food Science*, *60*(2), 234–236.
- Sun, H., Mu, T., Xi, L., Zhang, M., & Chen, J. (2014). Sweet potato (*Ipomoea batatas* L.) leaves as nutritional and functional foods. *Food Chemistry*, *156*, 380–389.
- Sun, J. B., Severson, R. F., Schlotzhauer, W. S., & Kays, S. J. (1995). Identifying critical volatiles in the flavor of BakedJewel Sweetpotatoes [*Ipomoea batatas* (L.) Lam.]. *Journal of the American Society for Horticultural Science*, *120*(3), 468–474.
- Tang, Y., Cai, W., & Xu, B. (2015). Profiles of phenolics, carotenoids and antioxidative capacities of thermal processed white, yellow, orange and purple sweet potatoes grown in Guilin, China. *Food Science and Human Wellness*, *4*(3), 123–132.
- Turkmen, N., Sari, F., & Velioglu, Y. S. (2005). The effect of cooking methods on total phenolics and antioxidant activity of selected green vegetables. *Food Chemistry*, *93*(4), 713–718.
- Wai, W. W., Alkarkhi, A. F. M., & Easa, A. M. (2010). Effect of extraction conditions on yield and degree of esterification of durian rind pectin: An experimental design. *Food and Bioprocess Technology*, *88*(2), 209–214.
- Wang, Y., & Kays, S. J. (2000). Contribution of volatile compounds to the characteristic aroma of baked jewel sweet potatoes. *Journal of the American Society for Horticultural Science*, *125*(5), 638–643.
- Wang, Y., & Kays, S. J. (2001). Effect of cooking method on the aroma constituents of sweet potatoes [*Ipomoea batatas* (L.) Lam.]. *Journal of Food Quality*, *24*(1), 67–78.
- Wang, S., Nie, S., & Zhu, F. (2016). Chemical constituents and health effects of sweet potato. *Food Research International*, *89*, 90–116.
- Wang, L., Xu, B., Wei, B., & Zeng, R. (2018). Low frequency ultrasound pretreatment of carrot slices: Effect on the moisture migration and quality attributes by intermediate-wave infrared radiation drying. *Ultrasonics Sonochemistry*, *40*, 619–628.
- Xiao, L., Lee, J., Zhang, G., Ebeler, S. E., Wickramasinghe, N., Seiber, J., & Mitchell, A. E. (2014). HS-SPME GC/MS characterization of volatiles in raw and dry-roasted almonds (*Prunus dulcis*). *Food Chemistry*, *151*, 31–39.
- Xu, B., Zhang, M., Bhandari, B., Cheng, X., & Sun, J. (2015). Effect of ultrasound immersion freezing on the quality attributes and water distributions of wrapped red radish. *Food and Bioprocess Technology*, *8*(6), 1366–1376.