Effect of brewing conditions on antioxidant properties of rosehip tea beverage: study by response surface methodology

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Revised: 26 July 2017 / Accepted: 9 August 2017 / Published online: 12 September 2017
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Abstract The aim of this study was to investigate the effects of brewing conditions (infusion time and temperature) on the antioxidant properties of rosehip tea beverage. The ascorbic acid content, total phenolic content (TPC), and ferric reducing antioxidant power (FRAP) of rosehip tea beverage were analysed. A two-factor and three-level central composite design was applied to evaluate the effects of the variables on the responses. The best quadratic models were obtained for all responses. The generated models were validated under the optimal conditions. At the optimal conditions, the rosehip tea beverage had 3.15 mg 100 mL⁻¹ of ascorbic acid, 61.44 mg 100 mL⁻¹ of TPC, and 2591 µmol of FRAP. The best brewing conditions for the rosehip tea beverage were found to be an infusion time of 6–8 min at temperatures of 84–86 °C.

Keywords Rosehip tea beverage · Response surface methodology · Ascorbic acid · Total phenolic · Ferric reducing antioxidant power

Introduction

Rosehips, which are fruits of the *Rosa* genus in the Rosaceae family have been used in food preparation and traditional medicine for a long time. Rosehips are traditionally used for the treatment of colds, the flu, and arthritis (Wenzig et al. 2008). Recently, rosehips have gained much attention due to their potential health

benefits. Rosehip species exhibit anti-inflammatory (De-liorman Orhan et al. 2007), antioxidant (Nadpal et al. 2016), antiproliferative (Jimenez et al. 2016), antiobesity (Ninomiya et al. 2007), and antidiabetic (Gholamhosseinian et al. 2009) properties. The biological activities of rosehips might be attributed to their composition. Rosehips are a good source of phytochemicals, such as vitamin C and E, phenolics, carotenoids, organic acids, and essential fatty acids (Barros et al. 2011).

Both fresh and dried rosehips are used in herbal tea (Wenzig et al. 2008). The consumption of herbal tea beverages has become more common because herbal teas include bioactive compounds that are beneficial to human health (Görgülü et al. 2016). Rosehip tea a good source of vitamin C, exhibiting antioxidant properties (Nojavan et al. 2008). In general, tea beverage is prepared by infusing tea with hot water. Brewing conditions might influence the extraction rate of antioxidant compounds from the tea to the tea beverage. Infusion time and temperature have been reported to be most critical parameters that affect the total phenolic content and antioxidant capacity of tea beverages (Lantano et al. 2015). The effect of brewing conditions on the composition of tea beverages is important for consumers since the beneficial effects of tea beverages are associated with the number of bioactive compounds extracted from the tea to the beverage. The effects of infusion time and water temperature on the antioxidant properties of black and green tea has been extensively studied (Hajlaghaalipour et al. 2016; Kelebek 2016; Sharpe et al. 2016). However, there is no available study on the effects of these brewing conditions on the antioxidant properties of rosehip tea beverage. Therefore, the aim of this study was to investigate the effects of brewing conditions (infusion time and temperature) on the

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antioxidant properties of rosehip tea beverage using response surface methodology (RSM). The ascorbic acid content, TPC, and FRAP were analysed.

Materials and methods

Experimental design

A two-factor and three-level central composite design was applied for this RSM study. The effects of two independent variables, infusion time and temperature, on the responses were investigated. The ranges studied were 70–90 °C for temperature (T), and 2–10 min for infusion time (t). Ten experimental settings (eight factorial points and two centre points) were generated using the Modde software (Umetrics, Sweden). The experimental design is presented in Table 1. Experiments were randomly run, and duplicate analyses were performed at each design point. The regression analysis, statistical significance, and response surface were analysed.

Tea beverage preparation

Dried rosehip was obtained from a local market. Rosehip was ground with a laboratory mill and screened through a 60-mesh sieve (IKA M20, Germany). After 2 g of tea was weighed and decanted into a 250-mL beaker, 100 mL of water at the studied temperature was added. After the tea was brewed at the experimental conditions in the water bath, the tea beverage was cooled and filtered through filter paper. The tea beverage was stored at 4 °C, and was analysed in 24 h.

Ascorbic acid content

Ascorbic acid content was determined according to the method described by Klein and Perry (1982) with some modifications. The tea beverage (2.5 mL) was extracted with meta-phosphoric acid (2.5 mL, 1%) using centrifugation at 5000 rpm for 30 min (Centurion Scientific, K2015R, England). The extract (0.5 mL) was mixed with 2,6-dichloroindophenol (70 mg/L, 4.5 mL). The absorbance was measured at 520 nm after being stored in the dark for 30 min. The calibration curve was prepared with ascorbic acid ranging from 0.008 to 0.03 mg mL⁻¹.

Total phenolic content

Total phenolic content (TPC) was estimated using Folin–Ciocalteu method. A 50-μL sample of the tea beverage was mixed with Folin–Ciocalteu reagent (500 μL), sodium carbonate (1 M, 400 μL), and water (4 mL). The absorbance was measured at 760 nm after 1 h. The calibration curve was prepared with gallic acid ranging from 0 to 1 mg mL⁻¹. TPC was expressed as mg of gallic acid per 100 mL of tea beverage.

Ferric reducing antioxidant power (FRAP) assay

First, fresh FRAP reagent was prepared by mixing the several solutions (10:1:1): acetate buffer solution (pH = 3.6), TPTZ solution (10 mM) in HCl (40 mM), and FeCl₃ (20 mM) solution. A 50-μL sample of the diluted tea beverage (1:2) was mixed with 3000 μL of FRAP reagent, and the absorbance was measured at 595 nm after 20 min. The results were expressed as micromoles of trolox.

Table 1 Central composite design and responses

Experiment no	Uncoded (coded) levels		Ascorbic acid (mg 100 mL ⁻¹)		TPC (mg 100 mL ⁻¹)		FRAP (μmol)	
	Time	Temperature	Observed	Predicted	Observed	Predicted	Observed	Predicted
1	2 (−1)	70 (−1)	2.98	2.95	52.8	53.7	2153.6	1981
2	2 (−1)	90 (1)	3.13	3.15	56.5	56.4	2937.9	2652
3	10 (1)	70 (−1)	2.92	2.95	58.5	57.4	2050.7	1951
4	10 (1)	90 (1)	3.15	3.15	59.1	60.1	2843.6	2622
5	6 (0)	80 (0)	3.24	3.23	61.3	61.4	2110.7	2301
6	2 (−1)	80 (0)	3.08	3.10	58.4	57.6	2415.0	2316
7	10 (1)	80 (0)	3.13	3.10	61.1	61.2	2522.1	2286
8	6 (0)	70 (−1)	3.07	3.08	57.3	57.5	2389.3	1966
9	6 (0)	90 (1)	3.29	3.28	61.1	60.2	2826.4	2637
10	6 (0)	80 (0)	3.21	3.23	60.9	61.4	2269.3	2301

Statistical analysis

Experimental design and data analyses were performed using Modde Pro (Umetrics, Sweden) software.

Results and discussion

Ascorbic acid content

The central composite design, the levels of factors, and the amount of ascorbic acid in the rosehip tea beverage under the experimental conditions are presented in Table 1. The ascorbic acid content of the rosehip tea beverage ranged from 2.92 to 3.29 mg 100 mL⁻¹.

The regression coefficients and *p*-values of the generated model are shown in Table 2. The response model equation for the ascorbic acid content can be written as follows:

$$Y = 3.229 + 0.002 t - 0.129 t^2 + 0.100 T - 0.054 T^2 \quad (1)$$

As shown in Table 2, temperature was the most significant independent variable. The first-order parameter of temperature (*T*), and the second-order parameters of temperature (*T* × *T*) and time (*t* × *t*) had significant effects on the ascorbic acid content. The linear term of temperature positively impacted the ascorbic acid content, whereas the quadratic terms of time and temperature negatively impacted the ascorbic acid content.

The analysis of variance (Table 3) revealed that the regression was significant (*p* < 0.01) and that the quadratic model showed no lack of fit value (*p* > 0.05). The correlation coefficient (*R*²) of the model was 0.98, indicating that the model explained 98% variability in the ascorbic acid content. The predicted values calculated from the model were found to be very close to the experimental values (Table 1). These findings indicated that the generated model can be used to explain the effects of the studied variables on the ascorbic acid content.

As shown in Fig. 1a, the ascorbic acid content of the rosehip tea beverage increased as time increased until the midpoint of response surface was reached. However, further increases in time decreased the ascorbic acid content. The ascorbic acid content showed an increasing trend with increasing temperature. The highest ascorbic content was obtained with infusion times between 5 and 8 min and at temperatures above 84 °C.

The ascorbic acid inside tea is diffused into the tea beverage during brewing. Water temperature is an important variable that affects the diffusion of ascorbic acid from the tea to beverage. The ascorbic acid content constantly increased under experimental conditions (70–90 °C). This finding might be attributed to an increase in the diffusion coefficient of ascorbic acid that accompanies increasing temperature. Arroqui et al. (2002) found that ascorbic acid's apparent diffusivity while blanching potatoes in water depended on temperature, and showed an Arrhenius-type equation. The apparent diffusivity coefficient was reported to increase with increasing temperature, ranging from 65 to 93 °C. An increase in the diffusivity coefficient was associated with the denaturation of cell membranes, which allows solutes like ascorbic acid to diffuse freely.

Infusion time also influenced the diffusion of ascorbic acid. Longer infusion times enhanced ascorbic acid extraction up to the maximum level, after which there was a reduction in this extraction. Ascorbic acid, which is a water-soluble vitamin, is the vitamin most sensitive to destruction. Enzymatic and thermal destruction reactions lead to ascorbic acid degradation. It can be easily oxidized in aqueous solutions (Burg and Fraile 1995; Yuan and Chen 1998). Longer infusion times might cause the thermal and enzymatic degradation of ascorbic acid.

Total phenolic content

Total phenolic content (TPC) of the rosehip tea beverage under the experimental conditions is shown in Table 1. The TPC of the tea beverage ranged from 52.8 to 61.1 mg 100 mL⁻¹.

Table 2 Regression coefficients and *p*-values

Factors	Ascorbic acid		TPC		FRAP	
	Regression coefficient	<i>p</i> Value	Regression coefficient	<i>p</i> -Value	Regression coefficient	<i>p</i> -Value
Mean	3.2292	0.001	61.4107	0.001	2301.41	0.001
Time (L)	0.0017	0.878	1.835	0.004	−15.0166	0.861
Time (Q)	−0.1286	0.001	−2.0114	0.015	55.7215	0.688
Temperature (L)	0.1000	0.001	1.3583	0.012	335.717	0.014
Temperature (Q)	−0.0536	0.031	−2.5214	0.007	195.021	0.204
Interaction (time × temperature)	0.0200	0.186	−0.795	0.102	2.1501	0.984

L linear, *Q* quadratic; Bold values are significant at 95% of confidence level

Table 3 Analysis of variance (ANOVA)

Factor	DF	Sum of square	Mean square	F-value	p-value
Ascorbic acid					
Regression	5	0.1137	0.0227	36.2075	0.002
Residual	4	0.0025	0.0006		
Lack of fit	3	0.0021	0.0007	1.5274	0.522
Pure error	1	0.0004	0.0004		
TPC					
Regression	5	62.8275	12.5665	22.1339	0.005
Residual	4	2.2708	0.5677		
Lack of fit	3	2.2096	0.7365	12.0525	0.208
Pure error	1	0.0612	0.0612		
FRAP					
Regression	5	777767	259256	9.6009	0.010
Residual	4	162021	27003.4		
Lack of fit	3	149444	29888.7	2.3765	0.450
Pure error	1	12577	12577		

Bold values are significant at 95% confidence of level

The regression coefficients and *p*-values of the generated model are shown in Table 2. The response model equation for the total phenolic content can be written as follows:

$$Y = 61.411 + 1.835 t - 2.011 t^2 + 1.358 T - 2.521 T^2 \quad (2)$$

The first-order parameters of temperature (*T*) and time (*t*), and the second-order parameters of temperature (*T* × *T*) and time (*t* × *t*) had significant effects on the TPC. The linear terms of temperature and time positively impacted the TPC, whereas the quadratic terms of time and temperature negatively impacted the TPC.

The analysis of variance (Table 3) revealed that the regression was significant (*p* < 0.01) and that the quadratic model showed no lack of fit value (*p* > 0.05). The correlation coefficient (*R*²) of the model was 0.97, revealing that the model explained 97% variability in the TPC. The predicted values calculated from the model were found to be very close to the experimental values (Table 1). These findings indicated that the generated model can be used to explain the effects of the studied variables on the TPC.

As shown in Fig. 1b, the TPC increased with increasing time up to a certain extent, and then changed slightly. The TPC of the rosehip tea beverage increased with increasing temperature until the midpoint of the response surface was reached. However, further increases slightly reduced the TPC. The highest TPC was obtained at times between 5.5 and 10 min and at temperatures between 77 and 86 °C.

First, the TPC of the rosehip tea beverage gradually increased with increasing infusion time. Our finding agreed with Kyle et al. (2007), who reported that the TPC of black

tea beverage increased with increasing infusion time, ranging from 3 to 10 min. The black tea was brewed for 3, 6 and 10 min and the highest TPC was obtained at the highest infusion time (Kelebek 2016). The TPC of green and black teas has been reported to increase with increasing infusion time, ranging from 0.5 to 10 min (Liebert et al. 1999). The TPC could be increased up to the maximum level since the solubility of phenolic compounds increased with an increase in time. The effect of infusion time on the solubility of green tea catechin was investigated by Labbe et al. (2006). They found that the catechin concentration increased up to certain level, and then remained constant, with increasing infusion time. The most abundant phenolic compound detected in rosehip (*Rosa canina*) was catechin (Demir et al. 2014). In our study, the TPC of the rosehip tea beverage remained constant after first increasing gradually. This finding might show that the rosehip tea beverage reached the maximum solubility level of phenolic compounds.

As the water temperature increased, the TPC increased up to certain level and then slightly decreased. Da Silveria et al. (2014) studied the effect of water temperature on the rutin content of mate tea beverages using RSM. The rutin content was found to increase with increasing temperature at lower levels, −1 and 0 (58.1–70 °C), and to decrease with increasing temperature at higher levels, 1 and 1.68 (81.9–90 °C). The catechin content of the green tea beverage prepared at different brewing temperatures (75, 85 and 95 °C) was investigated by Saklar et al. (2015). The epigallocatechin gallate (EGCG) was found to be the major catechin in Turkish green tea followed by epigallocatechin (ECG). The highest EGCG and ECG contents were

Fig. 1 Contour plots **a** ascorbic acid, **b** TPC, **c** FRAP

obtained at 85 °C. High temperature improves the extraction efficiency of components since the permeability of cell walls increases with heat treatment. However, higher

temperature can cause the degradation of phenolic compounds. In our study, the TPC content started to decline at temperatures above 86 °C after it reached its maximum

level. This finding can be attributed to the degradation of phenolic compounds.

FRAP

The FRAP values of the rosehip tea beverage under the experimental conditions are presented in Table 1. The FRAP values of the rosehip tea beverage ranged from 2051 to 2938 μmol trolox.

The regression coefficients and p -values of the generated model are shown in Table 2. The response model equation for the FRAP value can be written as follows:

$$Y = 2301.41 - 15.02 t + 335.72 T \quad (3)$$

As shown in Table 2, only first-order parameter of temperature (T) had significant effect on the FRAP value. The linear term of the temperature positively influenced the FRAP value.

The analysis of variance (Table 3) revealed that the regression was significant ($p \leq 0.01$) and that the generated model showed no lack of fit value ($p > 0.05$). The correlation coefficient (R^2) of the model was 0.83, indicating that the model explained 83% variability in the FRAP. The predicted values calculated from the model were found to be close to the experimental values. These findings indicated that the generated model can be used to explain the effects of the studied variables on the FRAP.

As shown in Fig. 1c, the FRAP value of the rosehip tea changed slightly with increasing time. It increased with increasing temperature. The highest FRAP value was obtained at temperatures above 84 °C.

As the water temperature increased, the FRAP values increased. This finding revealed that the antioxidant activity of the tea beverage increased with increasing brewing temperature. Some publications supported our finding. The FRAP value of the green tea was reported to increase with increasing temperature, ranging from 60 to 100 °C (Komes et al. 2010). The antioxidant activity of the white and green teas brewed at 90 °C was found to be higher than those brewed at 70 °C (Castiglioni et al. 2015). The black tea brewed at 100 °C showed higher antioxidant capacity than the tea brewed at 80 °C (Kelebek 2016). The green tea prepared at 90 °C was found to have five-fold higher FRAP values than the tea prepared at 20 °C (Langley-Evans 2000). The diffusion rate of antioxidant compounds from the tea to beverage might be enhanced by increasing temperature.

Validation of the models

The optimal conditions for the targeted responses (AA: 3 mg 100 mL⁻¹, TPC: 60 mg 100 mL⁻¹ and FRAP: 2500 μmol) were determined using the optimizer function of

Modde Pro (Umetrics, Sweden). The optimal conditions for the targeted responses were 10 min for infusion time, and 84 °C for infusion temperature. The experimental values (AA: 3.15 mg 100 mL⁻¹, TPC: 61.44 mg 100 mL⁻¹, and FRAP: 2591 μmol) were found to be very close to the predicted values (AA: 3.14 mg 100 mL⁻¹, TPC: 61.06 mg 100 mL⁻¹, and FRAP: 2508 μmol) obtained from the quadratic models, indicating that the models were adequate to predict the responses.

The visualization of contour plots revealed that the best brewing conditions for rosehip tea were 6–8 min and 84–86 °C to obtain the maximum ascorbic acid and polyphenols contents, and FRAP value.

Conclusion

The effect of brewing conditions (infusion time and temperature) on the ascorbic acid content, TPC, and FRAP value of the rosehip tea beverage was evaluated using RSM. Water temperature was found to be the most significant variable affecting the responses. The best brewing conditions for preparing rosehip tea beverage (2 g of tea in 100 mL water) were: a water temperature of 84–86 °C, and a brewing time of 6–8 min. Rosehip tea beverage is a good source of antioxidant compounds, such as ascorbic acid and phenolic compounds. When this beverage has been prepared under best brewing conditions, it can serve as a good source of antioxidant compounds in the human diet.

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