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Drying kinetics, total bioactive compounds, antioxidant activity, phenolic profile, lycopene and β -carotene content and color quality of Rosehip dehydrated by different methods

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ABSTRACT

This study aimed to determine the effect of different drying methods namely, hot air drying (HAD), freeze-drying (FD), vacuum drying (VD), and ultrasound-assisted vacuum drying (USVD) on drying kinetic, total bioactive compounds, lycopene and β -carotene, phenolic composition and color quality of rosehip. Drying times of the rosehips for USVD, VD, and HAD were 180, 300, and 1140 min, respectively, indicating that USVD significantly reduced drying time of rosehip. The results of total bioactive compounds, lycopene and β -carotene, phenolic composition showed that FD resulted the highest bioactive compounds, followed by VD, USVD, and HAD. DPPH and CUPRAC results showed that FD dried samples showed highest antioxidant activity than fresh and all dried samples while HAD showed lowest antioxidant activity. USVD and FD exhibited a lower ΔE value than HAD and VD, indicating that they had similar color properties to the fresh sample. USVD resulted in lower drying time and color change than VD and HAD, while USVD showed lower bioactive compounds retention than VD. This study suggested that USVD could be used as an alternative to HAD due to its lower drying time and higher bioactive compound and color retention than HAD.

1. Introduction

Rosehips (*Rosa canina*) is a reddish-color fruit belonging to the Rosaceae family that grows in Central Asian and Anatolian territories (Duru, Karadeniz, & Erge, 2011). While rosehip can be consumed as fresh, it can be processed into different products such as tea, jam, juice, marmalade (Erenturk, Gulaboglu, & Gultekin, 2005). Rosehip is rich in phenolic compounds, carotenoids, tocopherols, and vitamin C. Also, it contains A, B1, B2, K vitamins, calcium, phosphorus, potassium minerals, carbohydrates (pectin), and essential oils (Medveckienė, Kulaitienė, Jarienė, Vaitkevičienė, & Hallman, 2020). Rosehip has traditionally been used against many diseases because of its biological activities, which are immunosuppressive, antioxidant, anti-inflammatory, anti-arthritis, analgesic, anti-diabetic, cardioprotective, antimicrobial, gastroprotective, and skin ameliorative effects. Fresh rosehip has a short harvesting season, and it is sensitive to storage. Therefore, the fruit should be subjected to any preservation

method for its use all year round (Erenturk, Gulaboglu, & Gultekin, 2010).

Drying is one of the widely used preservation methods of fruit and vegetables. The drying process reduces water activity and the growth of microorganisms, lowers enzymatic activity and extends shelf-life at room temperature. Also, drying makes easier packaging, handling, and transportation by reducing the volume of material (Doymaz & Karasu, 2018). Hot air drying is still the most widely used method thanks to its economic benefit and ease of use, but if drying conditions are not controlled, hot air drying can cause degradation of thermo-sensitive compounds and loss of sensory and nutritional quality (Wojdylo et al., 2016). Freeze-dried products have high quality, high retention of nutrients and flavorings, and better rehydration properties, but freeze-drying is expensive and time-consuming (Barbosa et al., 2015). Due to these disadvantages of HAD and FD, alternative drying methods should be tried for drying fruit and vegetables in order to preserve their bioactive components.

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Vacuum drying is carried out without oxygen, and dehydration takes place at a mild temperature effectively. Therefore, the nutritional and sensory characteristics of products can be preserved. The vacuum drying might be combined with other methods to provide a more effective drying (Tekin & Baslar, 2018). Ultrasound-assisted vacuum drying (USVD) is one of the emerging methods applied in drying fruit and vegetables. Ultrasound contributes to vacuum drying to speed up heat and mass transfer. Via ultrasound power application, microscopic cavities are created on the tissues of fruit and vegetables. These microscopic channels make easier the transfer of the water found in internal tissues to the surface and provide evaporation. Thus, mass transfer increases and drying time reduces (Turkmen, Karasu, & Karadag, 2020). USVD has been used in the drying of some products which are red peppers (Tekin & Baslar, 2018), carrot slices (Chen, Guo, & Wu, 2016), nectarine (Souza da Silva et al., 2019), persimmon fruit (Kayacan et al., 2020), cherry laurel fruit (Turkmen et al., 2020) hawthorn fruit (Li, Wang, Wu, Wan, & Yang, 2020). However, ultrasound-assisted drying of rosehip cannot be found in the literature. This study aims to investigate the effects of different drying methods, namely HAD, VD, USVD, and FD, on drying kinetic, total bioactive content, antioxidant activity, individual phenolic compounds, β -carotene, lycopene content, and color change of rosehip fruits.

2. Materials and methods

2.1. Material

Fresh rosehip was supplied from Gumushane, Turkey, they were dried within 24 h after supplying and stored at 4 °C until drying processes. Rosehips with uniform shape, color, and size were selected for analysis. The initial moisture content of fresh rosehip was determined using an infrared moisture analyzer (Rad-wag, MA 50-R) and found as 39.10 ± 1.20 g/100g. Rosehips were drying as a whole. Dried rosehips were stored in a desiccator until analyses.

2.2. Methods

2.2.1. Drying procedure

Rosehips were dried by four different methods, namely, hot air drying (HAD), vacuum drying (VD), ultrasound-assisted vacuum drying (USVD), and freeze-drying (FD) as a whole. Drying processes for HAD, VD, and USVD were carried out at 50 °C. Drying temperature was chosen based on the bioactive compounds retention. In preliminary study, three different temperature (50, 60 and 70 °C) were applied and the temperature value showed higher TPC retention was chosen as drying temperature. HAD was performed at a constant air velocity of 1.3 m/s. The air velocity was measured by a Testo 440 vane probe anemometer (Lutron, AM-4201, and Taiwan). The airflow was applied horizontally through the surface of the rosehips. USVD was conducted according to the method described by (Başlar, Kılıçlı, Toker, Sağdıç, & Arici, 2014). The vacuum was regulated by a vacuum pump (EVP 2XZ-2C, Zhejiang, China) with 60 mbar ultimate pressure and 2 L/s pump speed in VD and USVD methods. FD was performed according to a standard program of freeze dryer (Martin Christ, Beta 1–8 LSC plus), and the samples were frozen at –80 °C overnight. Weight loss of rosehips was recorded at 60 min intervals during HAD, VD, and USVD. Drying processes were carried out until the moisture content of rosehips reached to 0.2 kg water/kg dry matter.

2.2.2. Mathematical modelling of the drying curve and effective moisture diffusivity (D_{eff})

In the present study, the obtained data were modeled by six thin-layer drying models, as illustrated in Table 1. According to this model, the moisture ratio was simplified to M/M_0 instead of $(M - M_e)/(M_0 - M_e)$ as the value of M_e is relatively small compared to M or M_0 , where M is the moisture content at time t , M_0 is the initial moisture content, and M_e

Table 1
Drying models described.

Models	Equation	Reference
Newton	$MR = \exp(-kt)$	Lewis (1921)
Page	$MR = \exp(-kt^n)$	Page (1949)
Henderson and Pabis	$MR = a \exp(-kt)$	Henderson and Pabis (1962)
Logarithmic	$MR = a \exp(-kt) + c$	Yagcioglu, Degirmencioglu, and Cagatay (1999)
Midilli and Kucuk	$MR = a \exp(-kt^n) + bt$	Midilli and Kucuk (2003)
Wang and Singh	$MR = 1 + at + bt^2$	Wang and Singh (1978)

is the equilibrium moisture content. The model parameters and R^2 values were calculated by performing nonlinear regression analysis using Statistica software program (StatSoft, Tulsa, USA). Model acceptability was determined based on R^2 and root mean square error (RMSE) values. Higher R^2 and lower RMSE values indicate good fitness of the established model. RMSE values were calculated according to following equation (Doymaz, 2012):

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^n (MR_{pre_i} - MR_{exp_i})^2 \right]^{1/2} \quad (1)$$

In this equation, MR_{pre} is the predicted moisture ratio, MR_{exp} is the experimental moisture ratio, N is number of observations and n is number of constants.

Effective moisture diffusivity (D_{eff}) of rosehip samples was calculated using Fick's second law which can be shown by the following equation:

$$\frac{M}{t} = \nabla [D_{eff}(\nabla M)] \quad (2)$$

This equation was adapted as the following equation assuming spherical shapes and unstable diffusion conditions:

$$MR = \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left(-n^2 \pi^2 \frac{D_{eff} t}{r^2}\right) \quad (3)$$

where D_{eff} is the effective moisture diffusivity (m^2/s), r is the half thickness of the rosehip (m), and n is the positive integer. For long drying periods, this equation can be simplified as Equation (4):

$$\ln(MR) = \ln\left(\frac{6}{\pi^2}\right) - \left(\pi^2 \frac{D_{eff} t}{r^2}\right) \quad (4)$$

D_{eff} values were calculated using the slope (K) of a straight line by plotting experimental drying data regarding $\ln(MR)$ versus time according to following equation:

$$K = \pi^2 \frac{D_{eff} t}{r^2} \quad (5)$$

2.2.3. Extraction procedure

Fresh and dried rosehips were extracted using methanol-water (50:50, v/v) solution with the 1:10 ratio. The mixture of rosehip and solution was homogenized at 10000 rpm for 2 min using an ultra-turrax (Daihan, HG-15D). Then the mixture was shaken for 2 h at room temperature. After 2 h, the mixture was centrifuged at 2800 g for 10 min. The supernatant was filtered through 0.45 μ m syringe filter (Kayacan et al., 2020).

2.2.4. Determination of total phenolic content

The total phenolic content (TPC) assay was determined according to modified method described by Singleton and Rossi (1965). 2.5 mL tenfold Folin Ciocalteu's phenol reagent, 2 mL 7.5 g/100g Na_2CO_3 , and 0.5 mL diluted methanolic rosehip extracts were mixed in a tube, and then this mixture was kept in the dark place for 30 min. The absorbance was read at 760 nm with a UV-Vis spectrophotometer (Shimadzu

UV-1800, Kyoto, Japan). The content of phenolics was expressed as mg gallic acid equivalents (GAE) per g of dry matter (DM) (mg GAE/g DM).

2.2.5. Determination of total flavonoid content

The total flavonoid content (TFC) was determined according to [Zhishen, Mengcheng, and Jianming \(1999\)](#). 1 mL of rosehip extracts was added to tubes that contain 4 mL distilled water. Then 0.3 mL 5 g/100 mL NaNO₂, 0.3 mL 10 g/100 mL AlCl₃ and 2 mL 1M NaOH were added to the tubes. To complete total volume to 10 mL, 2.4 mL distilled water was added, and the mixture was vortexed. The absorbance was recorded at 510 nm with a UV/VIS spectrophotometer. The flavonoids content was expressed as mg catechin equivalents (CE) per g of dry matter (mg CE/g DM).

2.2.6. Determination of antioxidant capacity

1,1-diphenyl-2-picrylhydrazyl (DPPH) and the copper-reducing antioxidant capacity (CUPRAC) methods were used to determine the antioxidant capacity extracts. 0.1 mL of extract was mixed with 4.9 mL 0.1 mmol/L DPPH•, which was soluted in methanol. After 30 min incubation in a dark place, absorbance was measured at 517 nm ([Singh, Chidambara Murthy, & Jayaprakasha, 2002](#)). CUPRAC assay was carried out as described by ([Apak, Güçlü, Ozyürek, & Karademir, 2004](#)). 1 mL of CuCl₂ (10 mmol/L), 1 mL neocuproine (7.5 mmol/L), and 1 mL NH₄Ac (1 mol/L), and then 1 mL distilled water was added to this mixture to complete the volume of the mix to 4.1 mL. Samples were incubated for 1 h in the dark, and the absorbance was measured at 450 nm. The results were expressed as mg Trolox equivalents (TE) per g of dry matter (mg TE/g DM).

2.2.7. Individual phenolic compounds

The content of individual phenolic compounds was determined using an HPLC-DAD method (Shimadzu Corp., Kyoto, Japan). The extracts obtained by methanol-water (50:50, v/v) solution with the 1:10 solid to liquid ratio were filtered by a 0.45-µm membrane filter. 1 mL of the filtered extract was injected in the HPLC system (LC-20AD pump, SPD20A DAD, SIL-20A HT autosampler, CTO-10ASVP column oven, DGU-20A5R degasser, and CMB-20A communications bus module; (Shimadzu Corp., Kyoto, Japan). Separations were carried out at 40 °C on a reversed-phase column (Intersil® ODS C-18, GL Sciences, Tokyo, Japan) with a 250 mm × 4.6 mm length, 5 µm particle size. The solvent A (distilled water with 0.1% (v/v) acetic acid) and solvent B (acetonitrile with 0.1% (v/v) acetic acid) were mobile phases. A gradient elution was 10% B (0–2 min), 10%–30% B (2–27 min), 30%–90% B (27–50 min) and 90%–100% B (51–60 min) and at 63 min returns to initial conditions. The flow rate was 1 mL/min. Chromatograms were recorded at 254–356 nm. The identification and quantitative analysis were conducted based on retention times and standard curves. The amounts of phenolic compounds for fresh and dried rosehip samples was presented as mg/L ([Kayacan et al., 2020](#)).

2.2.8. β-carotene and lycopene determination

The method reported by [Wright and Kader \(1997\)](#) with some modifications was used to carry out extraction of β-carotene and lycopene. For this aim, 2 g fresh and dried samples were mixed with 10 mL ethanol and homogenized with an ULTRA-TURRAX (Daihan, HG-15D) at 10,000 rpm for 3 min. Then 8 mL hexane was transferred to this solution and homogenized for 3 min. The final solution was centrifuged for 5 min at 6000 g. After centrifugation, the sediment was re-extracted with 5 mL of a saturated solution of NaCl and 8 mL hexane. Then, saponification of obtained supernatant was performed with 15 mL 10% methanolic KOH for 12 h. To remove KOH with an aqueous solution of NaCl (10 g/100 mL) and distilled water, this solution was transferred into a separatory system. The hexane phase was evaporated, and the residue was diluted by 2.5 mL acetone.

The content of β-carotene and lycopene were determined using an HPLC-DAD method. The analysis of β-carotene and lycopene was

conducted by HPLC coupled to a diode array (HPLC-DAD, Shimadzu Corp., Kyoto, Japan) with an HPLC system (LC-20AD pump, SPD20A DAD, SIL-20A HT autosampler, CTO-10ASVP column oven, DGU-20A5R degasser, and CMB-20A communications bus module); (Shimadzu Corp., Kyoto, Japan). Separations were carried out at 40 °C on Intersil® ODS C-18 reversed-phase column (250 mm × 4.6 mm length, 5 µm particle size). The distilled water, acetonitrile, and methanol (100/10/5) were selected as the mobile phase. A gradient elution were 10% B (0–2 min), 10%–30% B (2–27 min), 30%–90% B (27–50 min) and 90%–100% B (51–60 min) and at 63 min returns to initial conditions. The flow rate was 1 mL/min. Chromatograms were recorded at 450 nm. The retention times and standard curves were used to perform the identification and quantification of β-carotene and lycopene. The results were shown as mg/kg for fresh and dried samples.

2.2.9. Color properties

The color of fresh and dried rosehips was measured using a chromameter (Konica Minolta CR-400, NJ, USA). Color values were expressed as *L** (whiteness/darkness), *a** (redness/greenness), and *b** (yellowness/blueness) and the total color change ΔE of samples calculated as follow:

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (6)$$

A quantitative attribute of colorfulness Chroma, *C** was calculated following equation:

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (7)$$

2.2.10. Statistical analysis

Statistica software program (StatSoft, Inc., Tulsa, OK) was used to perform the statistical analysis. All the analyses were conducted in triplicate, and the standard error and mean value were presented. The one-way ANOVA was used to compare the mean values of the test results. Duncan's multiple comparison tests at 95% significance level were used to indicate the effect of different drying methods on bioactive compounds, phenolic profile change, β-carotene, lycopene, and color of rosehips. Pearson's coefficient of correlation was used to evaluate the relation between the antioxidant capacity and bioactive compounds.

3. Result and discussion

3.1. Effect of drying methods on drying time

Drying curves of rosehips samples were presented in [Fig. 1](#). Drying times of the rosehips were 180, 300 and 1140 min for USVD, VD and HAD, respectively. Moisture content of rosehips reduced exponentially with passed drying time. Drying time which required to reach 0.2 kg

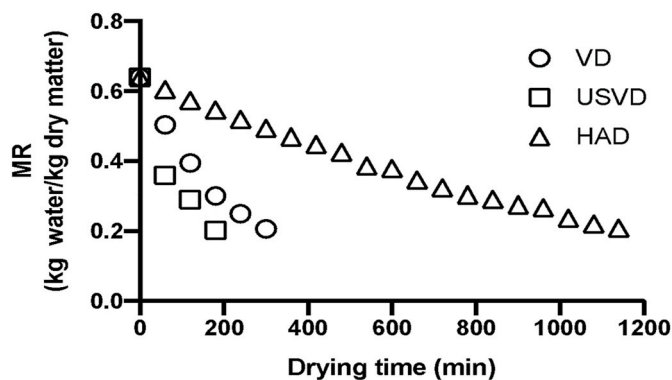


Fig. 1. Drying kinetic of rosehip (VD, vacuum drying; USVD, Ultrasound assisted vacuum drying; HAD, hot air drying; MR: moisture ratio).

water/kg DM moisture content differed significantly according to drying method. In HAD, external heat flux causes a slow increase of products' temperature, therefore moisture migration from interior is slow. This extends the time of drying (Wang et al., 2019). Vacuum process decreases the boiling point so that evaporation rate increases. Therefore, drying time of VD and USVD was approximately 4 times and 6.5 times less than HAD. It is clearly seen assisting ultrasound to vacuum provides a remarkable decrease in drying time. The decrease in drying time could be explained different ways: The cavitation effect generated by ultrasound can lead to a sudden and powerful expansion of moisture inside the samples, creating explosive force to create micro channels to reduce the diffusion resistance of the moisture (Liu, Zhu, Bai, You, & Yan, 2019; Li et al., 2020). Ultrasound can increase heat and moisture transfer rate by reducing internal viscosity and adhesion between microchannel and moisture (Azoubel, Baima, Amorim, & Oliveira, 2010; Chen et al., 2016). Also, previous studies have reported that USVD decreases the drying time (Chen et al., 2016; Li et al., 2020; Tekin & Baslar, 2018; Turkmen et al., 2020).

3.2. Modelling drying behavior and D_{eff} values

Six different models (Table 1) were applied to describe drying behavior of the rosehip. Table 2 showed R^2 value and model parameters of the selected models. R^2 values of the models were higher than 0.97, indicating that all drying models successfully describe drying behavior of the rosehip samples. Logarithmic and Midilli-Küçük models showed higher R^2 and lower RMSE value than that of other models for all drying methods. This model was selected as the best drying model to describe drying behavior of the samples. Drying kinetic coefficient of the k value was used to compare moisture transfer rate of the samples. k value was significantly affected from drying methods ($p < 0.05$). USVD showed the highest k value followed by VD and HAD for all drying models. D_{eff} values were shown in Fig. 2 and found as 8×10^{-10} , 3.5×10^{-9} and $6.78 \times 10^{-9} \text{ m}^2/\text{s}$ for HAD, VD and USVD, respectively. Similar results were reported by Márquez, De Michelis, and Giner (2006) for rosehip fruits. They found D_{eff} values ranged from 7.501×10^{-11} (50 °C) and 3.367×10^{-10} (80 °C) m^2/s for air forced circulation laboratory dryer. Also, the results showed that D_{eff} values were effected from the drying method. The highest D_{eff} value was found for USVD due to cavitation

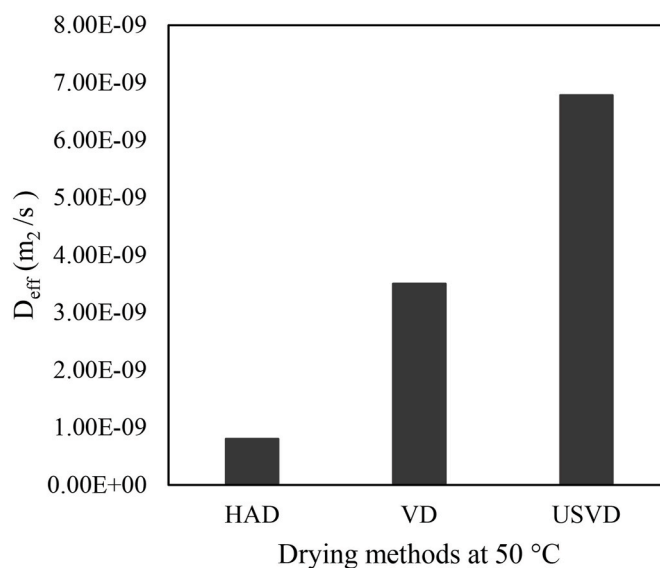


Fig. 2. Effective moisture diffusivity for drying methods at 50 °C.

effect as previously described. Higher D_{eff} values were found for USVD of green beans and red peppers (Tekin & Baslar, 2018; Tekin, Baslar, Karasu, & Kilicli, 2017). In a conclusion, USVD exhibited lower drying time and higher k and higher D_{eff} value compared to VD and HAD. These results indicated that ultrasound application with vacuum drying could increase drying rate of the rosehip.

3.3. Effect of drying methods on bioactive compounds of rosehip

Table 3 showed the effect of different drying methods on TPC, TFC, lycopene, β -carotene and AA of samples. TPC of fresh rosehip was found as 51.11 mg GAE/g DM. Koczka, Stefanovits-Bányai, and Ombódi (2018) extracted different rosehip species using water and ethanol. TPC values of rosehip changed with species and extraction solvent and were found as 150.8 mg–299.2 mg GAE/100 g DW for aqueous extracts and 255.9 mg–766.0 mg GAE/100 g DW for ethanolic extracts. In our study,

Table 2
Drying model coefficients for selected models.

Drying Methods	Models	Model coefficients	R^2	RMSE
HAD	Newton	k-1000	0.9978	0.0605
VD		0.928		
USVD		3.84		
HAD	Page	k-1000	0.9991	0.0386
VD		0.532 ^c		
USVD		6.88 ^b		
HAD	Henderson and Pabis	k-1000	0.9986	0.0280
VD		0.949 ^c		
USVD		3.76 ^b		
HAD	Logarithmic	k-1000	0.9993	0.0335
VD		0.66 ^c		
USVD		6.44 ^a		
HAD	Midilli and Kucuk	k-1000	0.9990	0.0291
VD		0.643 ^c		
USVD		5.32 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.695		
USVD		14.96 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
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VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114
VD		0.995		
USVD		33.67 ^a		
HAD	Wang and Singh	k-1000	0.9993	0.0339
VD		0.66 ^c		
USVD		2.51 ^b		
HAD	Wang and Singh	k-1000	0.9998	0.0114

Table 3
TPC, TFC, DPPH, CUPRAC Lycopene and β -carotene results of fresh and dried rosehip.

Bioactive properties	Fresh rosehip	Dried rosehip				<i>r</i>		
		HAD	VD	USVD	FD	TFC	DPPH	CUPRAC
TPC ¹	51.1 ± 0.3 ^a	31.5 ± 0.4 ^d	47.7 ± 1.2 ^b	44.7 ± 0.9 ^c	48.7 ± 1.3 ^b	0.9904	0.6700	0.9520
TFC ²	6.7 ± 0.1 ^a	4.4 ± 0.0 ^d	6.2 ± 0.1 ^b	5.9 ± 0.0 ^c	6.7 ± 0.1 ^a	1	0.7627	0.9516
DPPH ³	87.9 ± 0.7 ^a	60.6 ± 1.0 ^e	66.2 ± 0.0 ^c	65.8 ± 0.3 ^d	83.0 ± 0.2 ^b		1	0.7420
CUPRAC ⁴	304 ± 21.6 ^a	215 ± 12.8 ^c	295 ± 26.2 ^{ab}	256 ± 14.3 ^b	301 ± 5.3 ^a			1
Lycopene ⁵	24.1 ± 0.3 ^d	43.2 ± 4.5 ^c	122 ± 0.1 ^a	97.9 ± 5.4 ^b	116 ± 7.5 ^a			
β -carotene ⁶	10.6 ± 0.5 ^c	8.4 ± 0.1 ^e	11.9 ± 0.1 ^b	9.3 ± 0.4 ^d	13.0 ± 0.1 ^a			

HAD, hot air drying; VD, vacuum drying; USVD, ultrasound assisted vacuum drying; FD, freeze drying; different lowercase letters in the same line indicates differences between samples subjected to different drying methods ($p < 0.05$), *r*, Pearson coefficient of correlation.

¹ TPC expressed as mg GAE/g DM.

² TFC expressed as mg CE/g DM.

³ DPPH expressed as mg TE/g DM.

⁴ CUPRAC expressed as mg TE/g DM.

⁵ mg/kg.

⁶ mg/kg.

the effect of drying methods on TPC, TFC and AA was found to be significant ($p < 0.05$). TPC values of dried rosehips by VD, USVD and HAD decreased significantly when compared to fresh ones ($p < 0.05$). The high correlation was obtained between TFC and TPC results ($R > 0.9904$ (Table 1)). No significant difference was found between TFC values of fresh (6.67 ± 0.10 mg CE/g DM) and FD rosehips (6.66 ± 0.07 mg CE/g DM) ($p > 0.05$). The lowest TFC value was obtained from the samples dried by HAD (4.35 ± 0.02 mg CE/g DM). The highest and lowest TPC, TFC and AA values were found in FD and HAD, respectively. The higher retention of phenolic compounds in FD treated samples was also reported from the other studies (Chen et al., 2020; Golukcu, 2015; Kidon & Grabowska, 2021; Turkmen et al., 2020). The fact that the FD process takes place at a very low temperature and under vacuum conditions may have led to high retention of the phenolic compounds compared to other drying methods (Radojčin et al., 2021). HAD took place at a certain air velocity with a very long drying time. The irreversible oxidative reaction and thermal degradation might have resulted in degradation in phenolic compounds (Karasu et al., 2015) during long drying period. According to drying kinetic results, HAD showed lower drying kinetic and higher drying time than VD and USVD. Therefore, the bioactive compounds were subjected to less heat treatment during VD and USVD, therefore high retention in their level occurred. VD resulted in higher TPC, TFC and AA than USVD. The lower retention on phenolic compounds by ultrasound treatment were also reported from (Kroehnke et al., 2021; Nascimento, Mulet, Ascheri, Carvalho, & Carcel, 2016). According to results of some studies, USVD gave better protection for bioactive compounds when compared VD (Kayacan et al., 2020; Souza da Silva et al., 2019; Turkmen et al., 2020; Vieira da Silva Júnior, Lins de Melo, Batista de Medeiros, Pimenta Barros, & Azoubel, 2018). Different results from some studies could be explained by the different surface characteristics and low moisture content of rosehip. Rosehip has a waxy layer and this layer affects the flow of moisture from inside the fruit to its surface (Mabellini, Ohaco, Márquez, Lozano, & De Michelis, 2013). Although pores of rosehip opened during USVD, ultrasound could cause stress and damage bioactive compounds. The cavitation effect by ultrasound causes destruction in plant matrix and microcapillaries during drying of ultrasound assisted process. The low drying time observed in our study may not have been sufficient to separate phenolic compounds from the plant matrix. This situation may have reduced the effect of the ultrasound process on the extractability of phenolic compounds. The results of this study indicated that ultrasound assisted during vacuum drying could be less effective compared to VD preservation of bioactive compounds for drying of foods with a low moisture content and waxy layer. This outcome is important for future research about fruits or vegetables which have a waxy layer and low moisture content. In such products, USVD should be applied after or simultaneously some pretreatment process to increase bioactive compounds (Rahaman et al.,

2019). Although it resulted less retention in bioactive compounds than VD, USVD provided higher retention than HAD. Therefore, it can be used as an alternative method of drying rosehips due to its short drying time and acceptable bioactive protection. USVD could have the potential to be used more effectively with pretreatment application in whole drying of rosehip.

DPPH and CUPRAC values were used to calculate antioxidant activity of rosehips. FD had the highest DPPH and CUPRAC value than those of other drying methods. In addition, CUPRAC results of fresh (304 ± 21.63 mg TE/g DM), VD (294 ± 26.22 mg TE/g DM) and FD (300 ± 5.29 mg TE/g DM) values were not found different statistically ($p > 0.05$). In HAD, as in TPC and TFC, results of AA were found the lowest value. A high correlation was observed between TPC and antioxidant assays. Correlation coefficients of 0.67 and 0.95 were obtained between TPC and DPPH and CUPRAC, respectively. Correlation coefficients of 0.76 and 0.95 were found between TFC and DPPH and CUPRAC, respectively. These results showed that there was a high correlation between phenolic compounds and antioxidant capacity. The lower antioxidant capacity of HAD applied samples compared to other drying methods can be explained by higher degradation in phenolic compounds. The thermal treatment and oxidative reaction might have caused degradation of phenolic compounds during HAD process and reduced antioxidant capacity (Wojdyło et al., 2016).

Lycopene and β -carotene contents of the fresh and dried samples were shown in Table 3. Lycopene and β -carotene content of the fresh samples were found as 43.2–122.2 mg/kg and 8.4–13 mg/kg respectively. The effect of drying methods on lycopene and β -carotene was found to be significant ($p < 0.05$). VD and FD showed highest lycopene and β -carotene content while HAD had lowest lycopene and β -carotene content. USVD showed higher lycopene and β -carotene level than HAD while it resulted lower lycopene and β -carotene than FD and VD. Vacuum conditions and low drying time might have resulted higher retention of carotenoid compounds in USVD compared to HAD. The low drying time of USVD could lead to less degradation of lycopene and β -carotene, which are susceptible compounds to oxidative thermal degradation. USVD showed lower lycopene and β -carotene content than VD while USVD showed lower drying time. This results could be explained by cell disruption by cavitation effect. Higher degradation with application of ultrasound treatment was also reported from other studies (Kroehnke et al., 2021).

3.4. Effects of individual phenolic compounds

Table 4 described effect of drying methods on individual phenolic distributions of dried and fresh rosehip samples. Catechin and rutin were determined as most abundant phenolic compounds and their content varied as 105.4–394.4 mg/100g and 55.6–158.4 mg/100g respectively.

Table 4
Individual phenolic compounds fresh and dried samples.

Phenolic compounds (mg/100g)	Drying Methods				
	Fresh	USVD	VD	FD	HAD
Catechin	347 ± 16.9 ^b	255 ± 6.5 ^d	311 ± 9.8 ^c	394 ± 21.1 ^a	105 ± 4.3 ^e
	65.1 ± 2.8 ^a	48.6 ± 0.9 ^b	48.8 ± 0.9 ^b	66.7 ± 3.0 ^a	46.7 ± 0.1 ^c
Ellagic Acid	24.7 ± 0.4 ^a	14.6 ± 0.4 ^b	24.8 ± 1.0 ^a	24.2 ± 0.9 ^a	14.5 ± 0.7 ^b
	101 ± 9.6 ^a	96.8 ± 4.9 ^a	81.4 ± 4.0 ^b	97.0 ± 4.9 ^a	46.1 ± 2.7 ^c
Kaempferol	14.0 ± 0.5 ^b	20.6 ± 1.2 ^a	19.8 ± 1.5 ^a	21.3 ± 1.2 ^a	22.7 ± 1.8 ^a
	20.4 ± 1.0 ^c	22.3 ± 1.6 ^c	27.2 ± 2.0 ^b	33.8 ± 2.6 ^a	16.7 ± 0.3 ^d
Protocatechuic Acid	16.8 ± 1.7 ^{cd}	15.5 ± 1.0 ^d	32.3 ± 2.1 ^b	44.4 ± 3.5 ^a	18.6 ± 2.1 ^c
	87.4 ± 5.0 ^{ab}	73.2 ± 3.9 ^c	82.2 ± 4.3 ^b	93.2 ± 5.9 ^a	62.8 ± 3.7 ^d
Gallic Acid	137 ± 6.3 ^b	89.0 ± 3.8 ^d	106 ± 5.6 ^c	158 ± 8.5 ^a	55.6 ± 2.4 ^e
	50.1 ± 2.2 ^b	35.9 ± 2.1 ^d	41.3 ± 2.7 ^c	58.5 ± 2.4 ^a	27.9 ± 2.0 ^e
Syringic Acid	42.1 ± 2.3 ^b	27.4 ± 2.2 ^e	45.8 ± 3.1 ^b	61.2 ± 3.2 ^a	34.4 ± 2.7 ^c

HAD, hot air drying; VD, vacuum drying; USVD, ultrasound assisted vacuum drying; FD, freeze drying; different lowercase letters in the same line indicates differences between samples subjected to different drying methods ($p < 0.05$).

Catechin and rutin were also reported as the most abundant phenolic from (Elmastaş, Demir, Genç, Dölek, & Güneş, 2017) study. Among the phenolic acids, gallic acid, protocatechuic acid, ellagic acid were determined as major phenolic acid in methanolic rosehip extracts. Catechin, ellagic acid and gallic acid were reported as most abundant phenolic compounds by (Knez Hrnčić, Cör, Kotnik, & Knez, 2019). Similar to our study, ellagic acid was determined the most abundant phenolic acid by (Tumbas et al., 2012).

The effects of drying methods on individual phenolic compounds were found to be significant ($p < 0.05$). The distribution of individual phenolic contents was consistent with the TPC results. In general, a significant decrease was observed in the amount of phenolic components compared to the fresh sample in all drying methods, except for FD. The amounts of some phenolic compounds such as catechin, rutin and syringic acid in FD dried samples were higher than in fresh and other dried samples. In the samples dried with FD, the decrease in the amount of moisture content and the formation of micro capillaries with drying may have facilitated the extraction of some phenolic components. The low drying temperature without oxygen might not degradation in phenolic compounds in FD treated samples. Similar to TPC value, VD and USVD show a higher rate of individual phenolic compounds than HAD. The retention of the catechin as most abundant phenolic compound were 89%, 73% and 33% for VD, USVD and HAD respectively, indicating that most of the catechin degraded during HAD process. The retention of rutin showed similar trend with catechin and were found as 77, 65 and 40% for VD, USVD and HAD. In contrast to other phenolic compounds, kaempferol retention of the USVD was higher than that of VD. The fact that VD and USVD processes have low drying time and performed under vacuum may have caused a higher percentage of phenolic components compared to HAD. During the HAD process, a very high rate of degradation was observed in phenolic compounds with the effect of oxygen and temperature during the long drying period.

3.5. Effect of drying methods on color parameters

Color is one of the most important parameters affecting consumer expectations for dried samples. The L^* , a^* and b^* color parameters of the samples are shown in Table 5. L^* , a^* , b^* values of fresh rosehip were

Table 5
Color results of fresh and dried rosehip.

Color parameters	Fresh rosehip	Dried rosehip			
		HAD	VD	USVD	FD
L^*	42.5 ± 2.9 ^a	33.4 ± 2.3 ^c	39.2 ± 2.4 ^{ab}	37.1 ± 0.5 ^b	45.3 ± 0.8 ^a
a^*	24.7 ± 0.5 ^a	10.1 ± 0.8 ^c	24.2 ± 1.5 ^{ab}	22.5 ± 1.0 ^{ab}	21.9 ± 1.2 ^{ab}
b^*	13.3 ± 0.7 ^b	4.2 ± 1.0 ^c	19.0 ± 1.5 ^a	12.5 ± 1.9 ^b	13.5 ± 1.1 ^b
ΔE	-	19.4 ^a	6.7 ^b	5.9 ^c	5.8 ^c
C	28.0 ^b	11.0 ^e	30.8 ^a	25.7 ^c	23.3 ^d

HAD, hot air drying; VD, vacuum drying; USVD, ultrasound assisted vacuum drying; FD, freeze drying; different lowercase letters in the same line indicates differences between samples subjected to different drying methods ($p < 0.05$).

42.51, 24.68 and 13.25. As can be seen, the effects of different drying methods on L^* , a^* and b^* values were found to be significant ($p < 0.05$). While no significant difference was observed between the L^* values of fresh samples and that of FD and VD dried samples, a significant decrease was observed in the L^* values of the HAD and USVD samples compared to the fresh. The lowest L^* value was obtained from the sample dried with HAD. Non-enzymatic browning and enzymatic reactions during the long drying process may have caused more browning in the HAD-treated sample and thus a greater reduction in L^* value than other drying methods (Li et al., 2020). There was no significant difference between the a^* values of the USVD, VD, FD and the fresh samples. Rosehip is rich in carotenoids (Andersson, Rumpunen, Johansson, & Olsson, 2011; Medveckienė et al., 2020) and carotenoids are responsible for the color of rosehip. This results were consisted with the results obtained from lycopene and β -carotene analyses. According to lycopene and β -carotene results, HAD showed lower lycopene and β -carotene level than other methods. A significant decrease in a^* value of the samples dried with HAD can be explained by the degradation of carotenoid compounds that play an important role in the formation of the a^* value. According to Table 3, VD, USVD and FD showed higher retention of lycopene and β -carotene. Since there is less carotenoid degradation in VD, USVD and FD methods, no significant difference was observed in the a^* values of the samples. b^* value of the sample dried with VD increased while b value of the sample dried with the HAD decreased. USVD and FD did not change significantly b value. ΔE value is an important parameter showing the total color change in dried samples. The ΔE values of samples were found 5.76, 5.85, 6.66 and 19.39 for FD, USVD, VD and HAD. As can be seen, while ΔE value was similar in FD, VD and USVD samples, HAD was significantly higher than that of the other methods. Although there was no significant difference in VD, FD and USVD, it cannot be interpreted that these drying methods caused the same color change. Color parameters (L^* , a^* and b^*) showed different trend while total color change was not significant. While the L^* value in USVD decreased after drying, a^* value in VD and the L^* value in FD increased after drying process.

Pictures of the examples are shown in Fig. 3. As can be seen, the samples dried with HAD showed a rather dark color compared to the fresh sample. The images of the other 3 samples was similar to the fresh sample. In addition to the color values, more shrinkage was observed in the sample dried with HAD. This result shows that drying the rosehip with HAD is not suitable. While no shrinkage was observed in the sample dried with FD, partial shrinkage was observed in VD and USVD. These results showed that the samples dried with VD, USVD and FD exhibited similar color values and presented an image close to the fresh sample. The effect of drying temperature and air velocity on the color of rosehip was studied by (Koyuncu, Tosun, & Ustun, 2003). The dried samples obtained at all drying conditions showed lower L^* , a^* and b^* value than the of fresh ones.

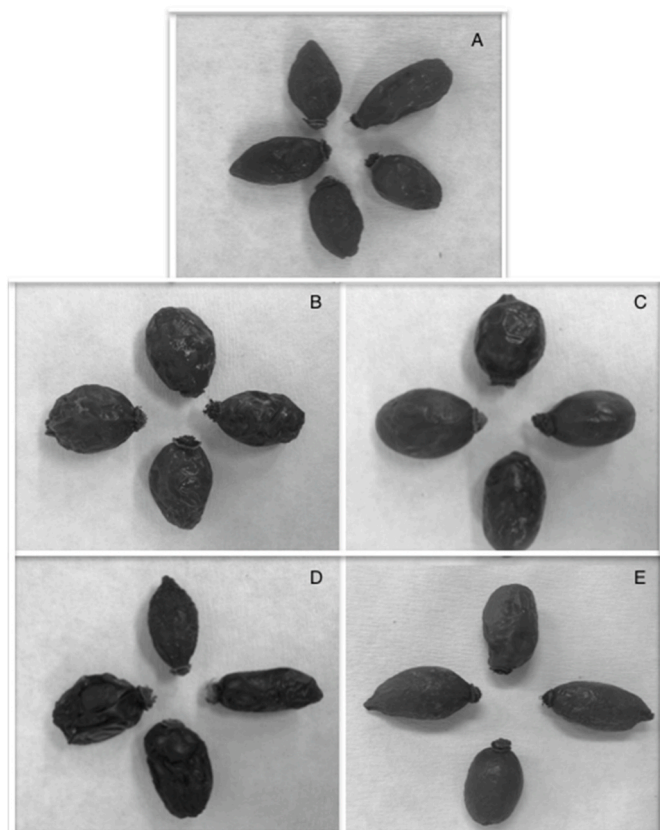


Fig. 3. The pictures of the fresh and dried samples A: fresh rosehip samples, B: vacuum dried (VD) rosehip samples, C: ultrasound assisted vacuum dried (USVD) rosehip samples D: hot air dried (HAD) raspberry samples, E: freeze dried (FD) rosehip samples.

4. Conclusions

In this study, the effects of different drying techniques on the drying kinetics and some quality properties of rosehip fruit were investigated. The combining of ultrasound to vacuum drying resulted in a remarkable decrease in drying time compared to VD and HAD. Logarithmic and Midilli and Kucuk models were selected as best drying models to describe drying behavior of the rosehip samples due to having higher R^2 value than that of other models. The highest phenolic compounds, lycopene and β -carotene retention was observed in FD dried samples. USVD significantly decreased compared to VD and HAD while USVD showed lower bioactive compounds retention than VD. This study suggested that ultrasound assisted to vacuum drying could be used as an alternative method to HAD due to its low drying time and higher bioactive retention than HAD.

CRedit authorship contribution statement

Berna Goztepe: Formal analysis, Investigation, Data curation. **Selma Kayacan:** Software, Formal analysis, Investigation, Data curation, Writing – original draft, Visualization. **Fatih Bozkurt:** Writing – original draft. **Merve Tomas:** Investigation, Data curation. **Osman Sagdic:** Supervision, Funding acquisition. **Salih Karasu:** Conceptualization, Methodology, Software, Investigation, Writing – review & editing, Visualization, Supervision, Funding acquisition.

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