Impact of Ultrasound Pretreatment and Temperature on Drying Kinetics and Quality Characteristics of Blood Orange: Comparison with Different Drying Methods

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Article

Impact of Ultrasound Pretreatment and Temperature on Drying Kinetics and Quality Characteristics of Blood Orange: Comparison with Different Drying Methods

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Abstract: This study aimed to investigate the impact of ultrasonic pretreatment vacuum drying (UAVD) and temperature on drying kinetics and qualitative attributes of blood oranges, in comparison to several drying methods: hot air drying (HAD), vacuum drying (VD), and freeze drying (FD). The drying kinetics and modeling, total phenolic content (TPC), antioxidant capability (assessed using DPPH and ABTS tests), individual phenolic profiles, vitamin C concentration, and color factors were meticulously examined. The HAD, VD, and UAVD procedures were conducted at 50, 60, and 70°C, resulting in reduced drying periods with increasing temperature. The integration of ultrasound markedly lowered drying durations. Eleven thin-layer drying models were utilized to recreate the drying process precisely. Among the desiccated blood orange slices, the greatest total phenolic content (TPC) was observed in freeze-dried samples (131.27 mg GAE/100g), followed by those dried using ultrasonic-assisted vacuum drying (UAVD) at 50°C (128.77 mg GAE/g DM). Dried blood orange slices have a vitamin C content of 29.79 to 49.01 mg/100. The drying process substantially impacted the color parameters L*, a*, and b*. These findings highlight the efficacy of ultrasound-assisted drying in decreasing drying duration while improving the retention of bioactive components in blood orange slices.

Keywords: blood orange; ultrasound-assisted vacuum drying; antioxidant capacity; phenolic profile; drying kinetics.

1. Introduction

Citrus fruits are commonly consumed around the world, primarily owing to their substantial nutritional value and related health benefits [1]. They constitute a significant portion of the world's fruit tree crops. During the 2022/23 marketing year, oranges accounted for 47% of global citrus production, totaling 48 million tons out of 100 million tons. In Turkey, citrus fruit production in 2023 reached 7.9 million tons, including 2.3 million tons of oranges, cultivated across 495 thousand hectares. The principal citrus-producing regions in Turkey are Antalya, Adana, Muğla, and Hatay [2].

The blood orange (*Citrus sinensis* (L.) Osbeck), commonly called pigmented or red-orange, comprises three primary cultivars: Moro, Tarocco, and Sanguinello. Unlike other orange varieties, the defining characteristic of blood oranges is the existence of red pigments in the flesh and, in some cases, the peel. These pigments, known as anthocyanins, develop in response to low night temperatures. As a result, blood oranges are predominantly cultivated in Mediterranean regions with subtropical climates characterized by high daytime temperatures and low nighttime temperatures [3&4]. Additionally, blood oranges are recognized for their elevated levels of total phenolics, anthocyanins, and flavones, which contribute to their superior antioxidant properties [5].

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A prevalent method for food preservation is drying, which provides benefits including prolonged shelf life, diminished packing and shipping expenses, and reduced weight and volume, thereby enhancing logistics. Additionally, drying enhances the retention of nutritional quality while lowering moisture content to levels that inhibit microbial growth [6&7]. Traditional hot-air convection drying is commonly utilized in the food industry because it is inexpensive and simple to implement. However, hot air drying has notable drawbacks, including adverse effects on food quality, environmental sustainability, and nutrient retention. Prolonged exposure to high temperatures during drying can significantly deteriorate bioactive chemicals and compromise their sensory attributes [8]. Novel drying methods that maximize energy economy while maintaining the quality characteristics of food items are therefore becoming more and more necessary [9].Among modern drying methods, freeze-drying is regarded as the most efficient approach to obtaining good-quality dried fruits and vegetables, as it preserves structural integrity and nutritional composition better than conventional methods. However, despite its advantages, freeze-drying is characterized by prolonged processing times and high operational costs, limiting its industrial application. As an alternative, VD has gained prominence for its ability to achieve faster drying rates at lower temperatures while operating in low-oxygen environments, thereby minimizing oxidative degradation of bioactive compounds. Advancements in drying technology have led to the evolution of integrated drying techniques that combine multiple methods to enhance efficiency, reduce costs, and improve sustainability. One such emerging approach is UAVD, which has gained attention for its potential to significantly accelerate drying while improving efficiency. This technique enhances the dehydration process by increasing the moisture transport rate without excessive thermal exposure. Water removal is accelerated by the cavitation effects produced by ultrasound, which help to create microchannels inside the food matrix. Furthermore, ultrasound promotes the extraction of tightly bound moisture by inducing localized pressure changes, all while maintaining relatively low temperatures, making it particularly beneficial for heat-sensitive food products [10]. A wide variety of food products including Asian pear [11], beef and chicken meats [12], carrot slices [13], green beans [10], nectarines [14], raspberry fruit [15], red peppers [16], salmon and trout fillets [17], *Schisandra chinensis* extract powder [18], papaya [19], and *Flos Sophorae Immaturus* [20] were effectively dried UAVD. The increasing adoption of UAVD underscores its significance as a modern dehydration technique that effectively addresses the limitations of conventional drying methods while ensuring superior preservation of food quality and nutritional integrity.

This study involved the drying of blood orange slices using HAD, VD, UAVD (at 50, 60, and 70°C), and FD methods. Several mathematical drying models were employed to examine drying kinetics and delineate the moisture removal behavior with time. Furthermore, the concentrations of TPC, DPPH, and ABTS, together with individual phenolic profiles, vitamin C levels, and color characteristics, were evaluated to ascertain the influence of various drying processes on the qualitative attributes of blood orange slices under varied temperature conditions.

2. Materials and Methods

2.1. Material

Blood oranges (Citrus sinensis (L.) Osbeck, cv. Sanguinello) were purchased in 2023 from an organic citrus farm located in Arsuz, Hatay, Turkey. Following procurement, the fresh oranges were transported to the Food Chemistry Laboratory at Yildiz Technical University and kept at 4°C till further processing. The initial percentage moisture of the fresh blood oranges was measured to be 83.10% ± 0.26% utilizing an infrared moisture analyzer (Rad-wag, MA 50-R). Prior to drying, the orange samples were thoroughly washed, wiped clean, and sliced into uniform 5-mm-thick sections. The dried slices were subsequently stored in a desiccator to preserve their structural integrity until further analysis.

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In this study, all the chemicals used for TPC, antioxidant capacity analyses (DPPH and ABTS), individual phenolic compounds, and vitamin C content were procured by Sigma-Aldrich (St. Louis, USA) and Merck (Darmstadt, Germany).

2.2. Methods

2.2.1. Drying Procedure

The drying of blood orange slices was conducted utilizing HAD, VD, and UAVD at 50, 60, and 70°C, and FD. During HAD, VD, and UAVD, the weight losses of orange slices were monitored at 30-minute intervals. The blood orange slices were subjected to drying until the moisture content attained 0.1 kg of water per kg of dry matter.

The HAD procedure was performed using a Testo 440 vane probe anemometer (Lutron, AM-4201, Taiwan) at a constant air velocity of 1.3 m/s. Horizontal airflow was applied over the surface of the slices throughout the drying process [11].

A vacuum drier (Daihan WOV-30, Gangwon-do, South Korea) was employed for

VD. A vacuum with an ultimate pressure of 60 mbar and a pump speed of 2 L/s was sustained using a vacuum pump (EVP 2XZ-2C, Zhejiang, China) [11].

In the UAVD technique, blood orange slices were subjected to a 30-minute ultrasonic water bath (Daihan, WUCD10H, South Korea) running at 100% amplitude, with a power intensity of roughly 1 W/cm², a frequency of 40 kHz, and a capacity of 10 L. Subsequently, blood orange slices were dehydrated using a vacuum dryer (DaihanWOV-30, Gangwon-do, Republic of Korea) [11].

FD was performed utilizing a defined protocol on a laboratory freeze drier (Martin Christ, Beta 1–8 LSC plus). The samples were cryopreserved at -80 °C and processed over a period of 72 hours [11].

2.2.2. Mathematical modeling

The moisture ratio (MR) was expressed as M_t/M_0 instead of $(M_t - M_e)/(M_0 - M_e)$, as the equilibrium moisture content (M_e) is negligible relative to the moisture content at time t (M_t) or the initial moisture content (M_0).

The drying rate (DR) curves were expressed as the weight loss per unit of time (g moisture/30 min). The drying rate (DR) of blood orange slices was determined by employing Eq. (1):

$$DR = \frac{\Delta M}{\Delta t} = \frac{M_{t+\Delta t} - M}{\Delta t} \quad (1)$$

where ΔM is the weight change, Δt is the time change, M_t and $M_{t+\Delta t}$ are the weight at time t and at time $t+\Delta t$, respectively. In this study, the weight change was measured every 30 min, so the Δt is 30 min.

The drying data collected during the experiments were analyzed by fitting them into eleven independent thin-layer drying models. Table 1 provides a comprehensive description of these models, which were assessed using nonlinear least squares regression analysis.

Table 1. Thin-layer drying models applied to the blood orange slices drying curves.

Model	Mathematical equation	Reference
Demir	y=a*exp(-k*x)^n+c	[21]
Henderson and Pabis	y=a*exp(-k*x)	[22]
Lewis	y=exp(-k*x)	[23]
Logarithmic	y=a*exp(-k*x)+c	[24]
Midilli and Kucuk	y=a*exp(-k*(x^n))+b*t	[25]
Modified Page	y=exp(-((k*x)^n))	[26]
Page	y=exp(-k*(x^n))	[27]

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Parabolic	y=a+b*x+c*x^2	[28]
Two-term exp	y=a*exp(-k*x)+(1-a)*exp(-k*a*x)	[29]
Wang and Singh	y=1+a*x+b*x^2	[30]
Weibull	y=exp(-(x^a/b^a))	[31]

The model parameters and R^2 values were ascertained by nonlinear regression analysis performed with the STATISTICA software (StatSoft, Tulsa, USA). The acceptability of each model was evaluated using R^2 and root mean square error (RMSE) values. A higher R^2 value and a lower RMSE value indicated a well-fitting model. RMSE values were computed by employing Eq. (2) [32]:

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2\right]^{1/2} \quad (2)$$

$$X^2 = \frac{\sum_{i-1}^{N}\left(MR_{pre,i} - MR_{exp,i}\right)^2}{N-m} \quad (3)$$

In Eq. 2&3, MR_{prei} represents the predicted moisture ratio, while MR_{expi} denotes the experimentally determined moisture ratio. N is the total count of observations, whereas m represents the quantity of constants used in the model.

The effective moisture diffusivity (D_{eff}) of the orange slices was calculated based on Fick's second law of diffusion (Eq. 4):

$$\frac{M}{t} = \nabla\left[D_{eff}\left(\nabla M\right)\right] \quad (4)$$

This equation was adapted to accommodate slab geometry and unsteady diffusion conditions, resulting in the following form:

$$MR = \frac{8}{\pi^2}\sum_{n=1}^{\infty}\frac{1}{(2n-1)^2}\exp\left(-(2n-1)^2\pi^2\frac{D_{eff}}{4L^2}t\right) \quad (5)$$

In Eq. 5, D_{eff} represents the effective moisture diffusivity (m^2/s), L is the thickness of the orange slice measured at regular intervals (m), and n is the constant derived from the thin-layer drying models. For extended drying periods, this equation can be simplified into the form presented in Eq. (6).

$$ln(MR) = ln\left(\frac{8}{\pi^2}\right) - \left(\pi^2\frac{D_{eff}}{4L^2}t\right) \quad (6)$$

The values of D_{eff} were determined utilizing the slope (K) of a straight line derived from plotting the experimental drying data as lnMR against time. This calculation was based on the following equation:

$$K = \frac{\pi^2 D_{eff}}{4L^2} \quad (7)$$

2.2.3. Total energy consumption

The drying tests were conducted with a digital energy meter (PeakTech 9035, Germany) attached to a socket to monitor the energy consumption. Upon completion of the dehydration process, the drying systems' total energy consumption was measured using the approach outlined by Tekin et al. [10].

2.2.4. Methods of analyses

2.2.4.1. Extraction procedure

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Fresh and dried orange samples were extracted with a methanol-water solution (50:50, v/v) at a ratio of 1:10 w/v. The orange sample mixture was homogenized at 10,000 rpm for 2 minutes using an ultra-turrax homogenizer (Daihan, HG-15D). Subsequent to homogenization, the mixture was agitated for 2 hours at 25 °C. Subsequent to shaking, the mixture was subjected to centrifugation at 2800 g for 10 minutes. The resultant supernatant was further filtered using a 0.45 µm syringe filter [33].

2.2.4.2. Determination of bioactive compounds

The TPC assay was carried out by Singleton and Rossi's modified procedure [34]. A UV–VIS spectrophotometer (Shimadzu UV-1800, Kyoto, Japan) measured the absorbance values at 760 nm. TPC was represented as mg GAE/g DM.

The analysis of DPPH was carried out by the methodology outlined by Brand-Williams et al. [35]. Absorbance was quantified at 515 nm, with results expressed as mg of Trolox equivalent (TE) per liter of material.

The ABTS radical cation scavenging capacity was evaluated using the technique established by Arnao et al. [36], with minor changes. The absorbance data was recorded at 734 nm, and outcomes were reported as mg TE/100 g DM.

2.2.4.3. Individual phenolic compounds

Individual phenolic compounds were analyzed by an HPLC system equipped with a diode array detector (SPD-M20A DAD, Shimadzu, Japan) in accordance with the methodology described by Turan et al. [37]. Chromatograms were monitored at wavelengths of 278, 320, and 360 nm, and the flow rate was maintained at 1 mL/min.

2.2.4.4. Vitamin C

The HPLC-DAD system (Shimadzu, Japan) was injected with 1 mL of the centrifuged blood orange juice after passing through a 0.45-µm filter (Millipore, Burlington, MA, USA). Supelco, Inc., Bellefonte, PA, USA, conducted the separation of ascorbic acid on a Supelcogel TM C 610H column (30 cm × 7.8 mm inner diameter) and a Supelguard column (5 cm × 4.6 mm) and detected the separation using a diode-array detector set at 210 nm. Pure standards of ascorbic acids were quantified using standard curves. The ascorbic acid concentrations are reported as milligrams per 100 grams of sample [38].

2.2.4.5. Color

The color of both fresh and dried orange slices was evaluated utilizing a chromameter (Konica Minolta CR-400, NJ, USA). The color parameters have been identified as L* (lightness/darkness), a* (redness/greenness), and b* (yellowness/blueness). The total color change (ΔE) of the slices was determined using the subsequent formula:

$$\Delta E = \sqrt[2]{\Delta L^2 + \Delta a^2 + \Delta b^2} \quad (7)$$

2.2.5. Statistical analysis

The statistical analysis was carried out using the Statistica software program (StatSoft, Inc., Tulsa, OK). All experiments were conducted in triplicate, and the results were expressed as the mean values along with their standard errors. A one-way ANOVA was performed to compare the mean values of the test results. Duncan's multiple comparison test, at a 95% confidence level, was applied to evaluate the impact of different drying methods on bioactive compounds, changes in the phenolic profile, vitamin C levels, and the color characteristics of the orange samples.

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3. Results

3.1. Drying kinetics

Fresh blood orange slices had an initial moisture percentage of 83.10% ± 0.26%. On a wet basis, the slices were dried until they had a final moisture percentage of 10%. Drying was performed using HAD, VD, and UAVD methods at temperatures of 50, 60, and 70 °C, as well as through FD. Figure 1 shows the pictures of fresh and dried blood oranges with HAD, VD, UAVD, and FD. The samples that were dried using HAD were found to have less volume.

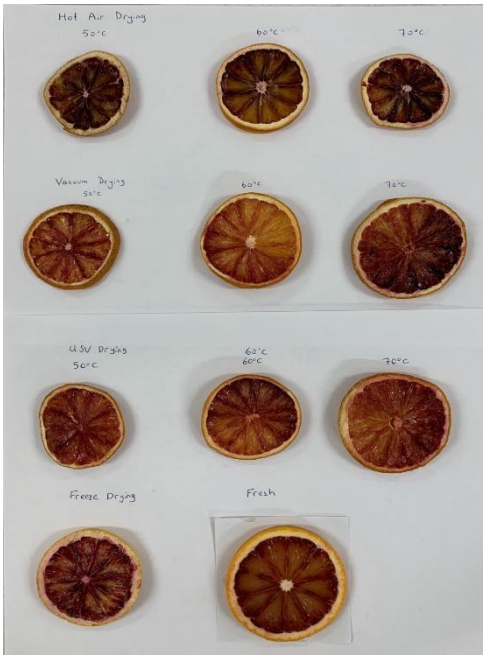

Figure 1. Fresh and dried blood oranges with HAD, VD, UAVD, and FD.

Figure 2 indicates the experimental data for the moisture ratio (MR) vs time curves of blood oranges dried with HAD, UAVD, and VD methods. Regardless of the drying methods, the moisture ratio (MR) continuously diminished during drying period. There is no constant rate period in these curves, only a decreasing rate period is observed. The MR values were utilized to assess the eleven models delineated in Table 1.

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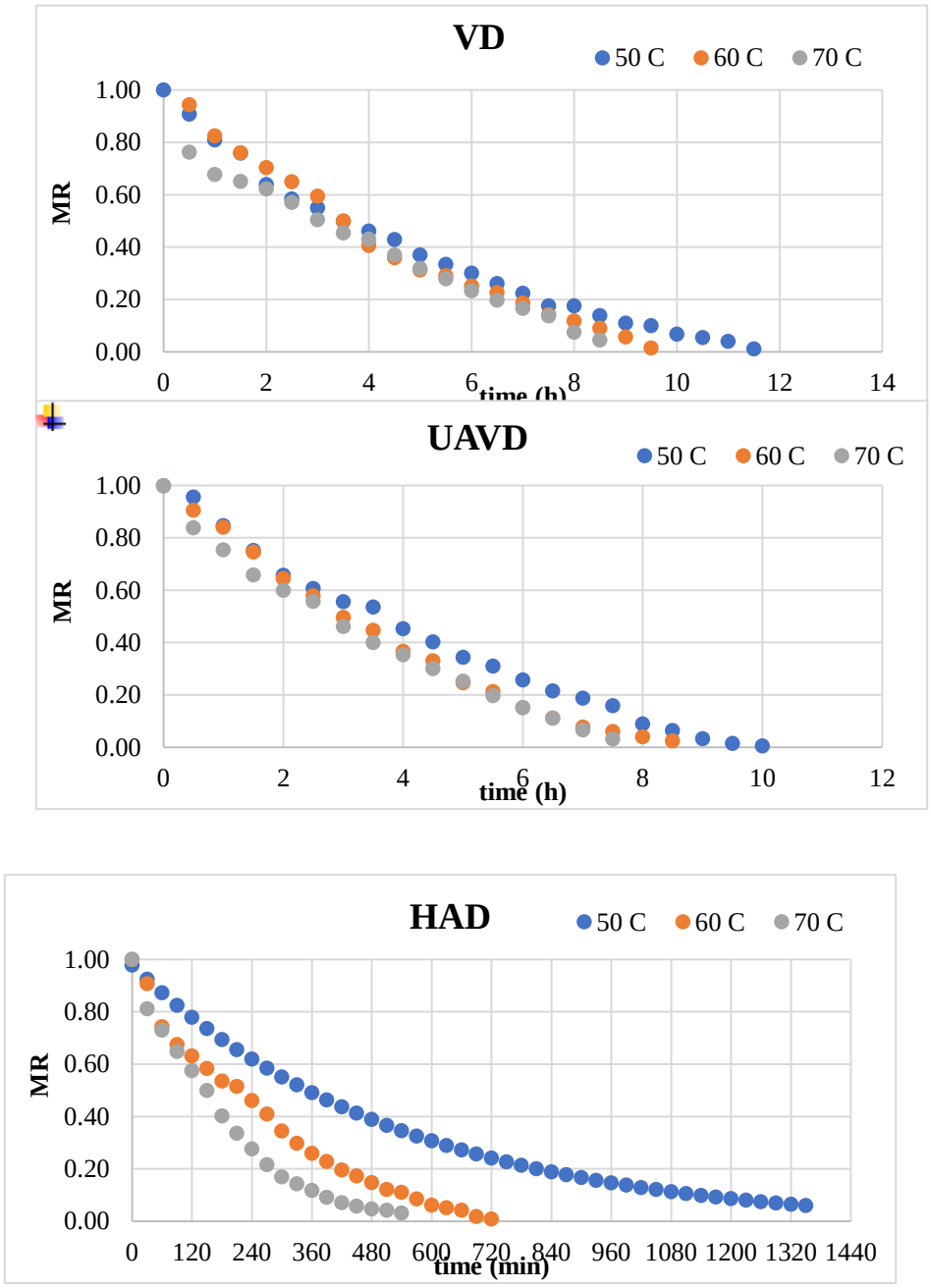

Figure 2. Moisture ratio (MR) vs time (h).

Table 2 displays the predicted model parameters and the statistical metrics for blood orange slices. The model that most accurately represented the thin-layer drying kinetics was selected based on the greatest R^2 values and the lowest RMSE and χ^2 values. Table 2 indicates that the Logarithmic model was the most effective for the HAD, VD, and UAVD approaches, with R2 values ranging from 0.997 to 0.998, 0.999 to 1.000, and 0.999 to 1.000, respectively. The k values for the Logarithmic model were established as 0.131, 0.166, and 0.330 for 50, 60, and 70 °C for HAD drying, and 0.156, 0.156, and 0.152 for VD, respectively. Consequently, a vacuum facilitates the transfer of water from the inside to the exterior due to the elevated water vapor pressure at reduced temperatures. Akdas and Baslar [39] identified the logarithmic model as the most effective model for Mandarin. The remaining 10 models exhibited high determination coefficients (R^2) ranging from 0.979 to 0.998 for HAD, from 0.983 to 1.000 for VD, and from 0.982 to 1.000 for UAVD methods.

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Table 2. Calculated parameters and statistical model parameters according to HAD, VD, and UAVD.

Model	Parameters	HAD			VD			UAVD		
		50 °C	**60 °C**	**70 °C**	**50 °C**	**60 °C**	**70 °C**	**50 °C**	**60 °C**	**70 °C**
Demir et al.	**a**	0.918	1.111	1.175	1.149	1.391	1.352	1.149	1.391	2.004
	k	0.330	-0.411	-0.559	-0.419	-0.436	-0.389	-0.419	-0.436	-0.301
	n	0.396	-0.403	-0.480	-0.372	-0.358	-0.392	-0.372	-0.358	-0.304
	c	0.049	-0.144	-0.086	-0.164	-0.371	-0.390	-0.164	-0.371	-0.989
	R^2	0.983	0.997	0.998	0.999	0.999	0.998	0.999	0.999	0.999
	RMSE	0.0455	0.0212	0.0192	0.0165	0.0152	0.0161	0.0137	0.0195	0.0114
	χ^2	0.0023	0.0005	0.0005	0.0003	0.0003	0.0003	0.0002	0.0005	0.0002
Henderson and Pabis	**a**	0.943	1.006	1.015	1.027	1.078	1.013	1.027	1.078	1.087
	k	0.112	0.229	0.330	0.221	0.286	0.286	0.221	0.286	0.272
	R^2	0.982	0.993	0.995	0.994	0.987	0.998	0.994	0.987	0.982
	RMSE	0.0466	0.0343	0.0286	0.0470	0.0489	0.0399	0.0314	0.0356	0.0576
	χ^2	0.0023	0.0013	0.0009	0.0025	0.0027	0.0018	0.0011	0.0014	0.0038
Lewis	**k**	0.120	0.227	0.325	0.215	0.265	0.282	0.215	0.265	0.248
	R^2	0.979	0.993	0.995	0.993	0.983	0.990	0.993	0.983	0.976
	RMSE	0.0499	0.0344	0.0290	0.0532	0.0565	0.0402	0.0327	0.0448	0.0666
	χ^2	0.0025	0.0012	0.0009	0.0030	0.0034	0.0017	0.0011	0.0021	0.0047
Logarithmic	**a**	0.918	1.111	1.075	1.149	1.391	1.352	1.149	1.391	2.004
	k	0.131	0.166	0.330	0.156	0.156	0.152	0.156	0.156	0.091
	c	0.049	0.144	0.186	-0.164	-0.371	-0.390	-0.164	-0.371	-0.989
	R^2	0.983	0.997	0.998	0.999	0.999	0.998	0.999	0.999	0.999
	RMSE	0.0455	0.0212	0.0192	0.0165	0.0152	0.0161	0.0137	0.0195	0.0114
	χ^2	0.0022	0.0005	0.0004	0.0003	0.0003	0.0003	0.0002	0.0005	0.0002
Midilli et al.	**a**	0.959	1.035	1.314	0.966	0.969	4.663	0.966	0.969	0.968
	k	0.177	0.191	0.238	0.149	0.135	0.054	0.149	0.135	0.108
	n	0.809	0.842	1.203	1.194	1.449	0.723	1.194	1.450	1.559

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	b	-0.00004	0.00003	0.708	0.0000004	-0.000015	2.890	0.0000004	-0.00001	0.00006
	t	-0.00004	0.00003	0.446	0.0000004	-0.000015	-1.268	0.0000004	-0.00001	0.00006
	R^2	0.986	0.998	0.998	0.996	0.998	1.000	0.996	0.998	0.997
	RMSE	0.0406	0.0299	0.0183	0.0314	0.0192	0.0313	0.0243	0.0176	0.0233
	χ^2	0.0018	0.0011	0.0005	0.0013	0.0005	0.0014	0.0007	0.0004	0.0008
Modified Page	**k**	0.123	0.224	0.319	0.211	0.259	0.279	0.211	0.259	0.247
	n	0.845	1.083	1.127	1.131	1.374	1.140	1.131	1.374	1.464
	R^2	0.986	0.994	0.997	0.996	0.998	0.993	0.996	0.998	0.997
	RMSE	0.0411	0.0319	0.0222	0.0320	0.0213	0.0343	0.0256	0.0207	0.0255
	χ^2	0.0018	0.0011	0.0004	0.0011	0.0005	0.0013	0.0007	0.0005	0.0007
Page	**k**	0.170	0.198	0.276	0.172	0.186	0.233	0.172	0.156	0.129
	n	0.845	1.083	1.127	1.131	1.374	1.140	1.131	1.374	1.464
	R^2	0.986	0.994	0.997	0.996	0.998	0.993	0.996	0.998	0.997
	RMSE	0.0411	0.0319	0.0222	0.0320	0.0213	0.0343	0.0256	0.0207	0.0255
	χ^2	0.0018	0.0011	0.0006	0.0011	0.0005	0.0013	0.0007	0.0005	0.0007
Parabolic	**a**	0.877	0.937	0.957	0.957	1.012	0.951	0.957	1.012	1.014
	c	0.002	0.006	0.014	0.006	0.010	0.009	0.006	0.010	-0.178
	b	-0.072	-0.148	-0.224	-0.146	-0.198	-0.186	-0.146	-0.198	0.006
	R^2	0.970	0.996	0.998	0.998	0.999	0.998	0.998	0.999	0.999
	RMSE	0.0598	0.0251	0.0163	0.0184	0.0106	0.0187	0.0191	0.0241	0.0104
	χ^2	0.0038	0.0004	0.0001	0.0004	0.0001	0.0004	0.0004	0.0007	0.0001
Two Term Exp	**a**	0.219	1.544	1.625	1.621	1.890	1.636	1.621	1.889	1.943
	k	0.276	0.413	0.435	0.273	0.394	0.364	0.273	0.394	0.386
	R^2	0.988	0.994	0.997	0.996	0.997	0.993	0.996	0.997	0.995
	RMSE	0.2646	0.1535	0.0905	0.0341	0.0257	0.0332	0.0250	0.0217	0.0320
	χ^2	0.0016	0.0010	0.0005	0.0013	0.0007	0.0013	0.0007	0.0005	0.0012
Wang & Singh	**a**	-0.094	-0.169	-0.242	-0.160	-0.193	-0.212	-0.160	-0.193	-0.171
	b	0.002	0.007	0.015	0.007	0.009	0.011	0.007	0.009	0.005
	R^2	0.955	0.993	0.997	0.996	0.999	0.996	0.996	0.999	0.999

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	RMSE	0.0735	0.0339	0.0227	0.0185	0.0115	0.0264	0.0246	0.0245	0.0117
	χ^2	0.0057	0.0012	0.0006	0.0004	0.0001	0.0008	0.0007	0.0007	0.0002
Weibull	**a**	0.845	1.083	1.127	1.131	1.374	1.140	1.131	1.374	1.464
	b	8.114	4.456	3.138	4.732	4.865	3.585	4.733	3.865	4.042
	R^2	0.986	0.994	0.997	0.996	0.998	0.993	0.996	0.998	0.997
	RMSE	0.0411	0.0319	0.0222	0.0320	0.0213	0.0343	0.0256	0.0207	0.0255
	χ^2	0.0018	0.0011	0.0006	0.0011	0.0005	0.0013	0.0007	0.0005	0.0007

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Table 3 indicates the drying time, drying rate, effective moisture diffusivity, and total energy consumption of the blood orange slices. Drying times were recorded as 22.5 hours, 12.5 hours, and 9 hours for HAD; 11.5 hours, 9.5 hours, and 8.5 hours for VD; and 10 hours, 8.5 hours, and 7.5 hours for UAVD at 50, 60, and 70 °C, respectively. A reduction in drying time was observed with increasing temperatures. Further analysis revealed that ultrasonic pretreatment significantly decreased the drying period. The effectiveness of ultrasound in minimizing drying durations has also been reported in other studies involving various fruits [37, 40, 41]. The gradual increase in the product's temperature throughout the hot air-drying process, due to external heat transfer, impedes internal moisture migration, thereby extending the drying duration. In contrast, vacuum drying decreases pressure, which reduces the water boiling point in the food, hence increasing the surface evaporation rate. The combination of ultrasound with vacuum has been shown to significantly reduce drying times. This effect is attributed to cavitation, which creates microscopic voids within the structure of fruits and vegetables, facilitating easier water removal and thereby shortening the drying process. The secondary consequence is the mechanical impact induced by ultrasound, which diminishes the adhesion between moisture and the microtube, hence expediting moisture evacuation [42]. Furthermore, ultrasonic treatment can diminish internal viscosity and enhance the heat transfer coefficient [13]. The last impact of ultrasound is the thermal effect, which induces a marginally accelerated temperature rise, advantageous for the internal diffusion and evaporation of moisture [40].

Table 3. Drying time, drying rate, effective moisture diffusivity, and total energy consumption of the blood oranges.

Drying Method	Temperature (°C)	Drying time (h)	DR* (h^{-1})	D_{eff}** (m^2/s)	Total Energy consumption (kW·h)
HAD	50	22.5^{Aa}	0.044	9.08×10^{-10}	0.163
	60	12.0^{Ab}	0.083	2.32×10^{-9}	0.209
	70	9.0^{Ac}	0.108	2.82×10^{-9}	0.266
VD	50	11.5^{Ba}	0.082	2.60×10^{-9}	0.196
	60	9.5^{Bb}	0.115	2.76×10^{-9}	0.235
	70	8.5^{Bc}	0.130	2.96×10^{-9}	0.286
UAVD	50	10.0^{Ca}	0.104	2.20×10^{-9}	0.298
	60	8.5^{Cb}	0.118	2.90×10^{-9}	0.333
	70	7.5^{Cc}	0.135	2.99×10^{-9}	0.372

*DR: Drying rate and **D_{eff}: Effective moisture diffusivity. Different lowercase letter displays significance of the parameters. Different uppercase letter displays significance of the parameters($p<0.5$). .

The drying of various foods under constant conditions typically results in curves with distinct shapes during the falling rate period [43]. Damage to the cell structure of foods may result in deviations during the constant drying rate period [10]. Ultrasonic treatment enhances the drying rate by leveraging the beneficial impacts of heating through attenuation and adsorption, in conjunction with the mechanical effects of pressure waves [10, 12, 13]. Table 3 indicates that the drying rate values for the HAD, VD, and UAVD methods varied with drying temperature. The drying rate values obtained through the UAVD method, as presented in Table 3, exceeded those of the HAD and VD methods during the falling rate period, attributable to the beneficial effects of ultrasound. The drying rate values escalated with all drying techniques as the temperature increased. A shortened period is

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essential for the material to attain equilibrium moisture content. Figure 3 illustrates the drying rate vs kg water/kg drymatter.

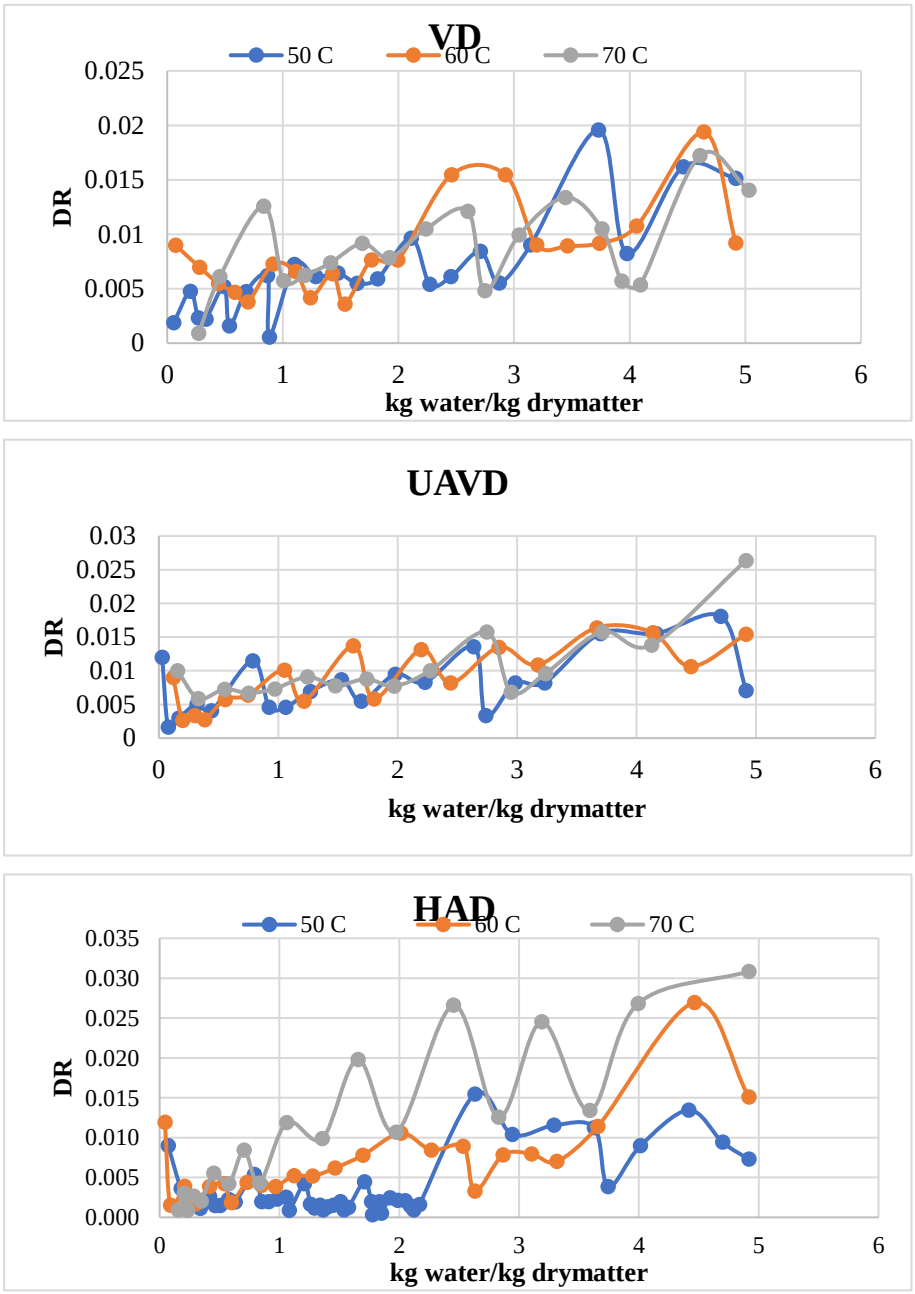

Figure 3. Drying rate vs kg water/kg drymatter.

In the current study, the D_{eff} rose as the temperature rose in all of the drying processes. The D_{eff} values of the HAD, VD, and UAVD dried samples varied from $9.08*10^{-6}$ to $2.82*10^{-5}$ m^2/s, from $2.60*10^{-5}$ to $2.96*10^{-5} m^2/s$, from $2.20*10^{-5}$ to $2.99*10^{-5}$, respectively. Table 3 displays the D_{eff} values for the HAD-, VD-, and UAVD-dried blood orange slices at 50, 60, and 70 °C. The D_{eff} values of the UAVD-dried samples are higher than those of the HAD-dried and VD-dried blood oranges. Table 3 presents the D_{eff} values for the HAD, VD, and UAVD methods at 50, 60, and 70 °C. The D_{eff} values of the UAVD-dried samples exceed those of the HAD-dried and VD-dried blood oranges. The data in Table 3 suggest that D_{eff} increases as the temperature rises, which is because the hydration content of the blood oranges decreases due to the rapid evaporation of water molecules. As the temperature increased, Huang and Chen [44] noted that the D_{eff} of sewage sludges increased.

Energy consumption varies with drying methods and temperature, and it decreases as the temperature increases. This study found that total energy consumption varied from 0.163 to 0.266

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kWh with HAD, from 0.196 to 0.286 kWh with VD, and from 0.298 to 0.372 kWh with the UAVD method. The total energy consumption of samples dried using the UAVD method exceeded that of other drying methods; however, the drying times with UAVD were shorter, suggesting that UAVD drying may be more economical.

3.2. Total phenolic content and antioxidant capacity

Citrus fruits are abundant in phenolic acids and flavonoids, two principal categories of natural antioxidants that underlie their functional qualities. The differences in flavonoid content in fruits are primarily due to biological factors [45]. These secondary metabolites serve several functions in the plant; specifically, in the fruit, they are linked to color, sensory attributes (flavor, astringency, texture), nutritional properties, and antioxidant activity [46].

TPC values and antioxidant capacity values determined by DPPH and ABTS methods of fresh blood orange slices and blood orange slices dried with HAD, VD, and UAVD at 50, 60, and 70 °C, as well as through freeze-drying are shown in Table 4. The TPC value of the fresh blood orange slice was 154.25 mg GAE/100g. The drying techniques and temperatures substantially influenced the total bioactive phenolic compounds and antioxidant activity levels. The FD dried blood orange slices have the highest TPC value (131.27 mg GAE/100g) within the dried blood orange slices. The TPC result of the samples dried by UAVD at 50 °C (128.77 mg GAE/100g DM) is comparable to that of the FD-dried samples (131.27 mg GAE/100g DM)($p<0.05$). The preservation of TPC is due to the low temperature and vacuum conditions inherent to the FD process. Tekin-Cakmak et al. [15] and Goztepe et al. [33] indicated that some red fruits, when dried using similar procedures, had the greatest TPC values in freeze-dried samples. The TPC of dried blood orange slices diminished as a result of thermal degradation at rising temperatures throughout the drying process. For UAVD, TPC values decreased from 128.77 mg GAE/g DM to 65.25 mg GAE/g DM by increasing the temperature from 50 °C to 70 °C. Similarly, research on Tunisian eggplants examined how different drying techniques impacted their drying properties and bioactive compounds. Chouaibi et al. [47] established that both freeze-drying and ultrasound-assisted drying reduced product deterioration, corroborating results from analogous research. The elevated TPC value of UAVD-dried blood orange slices has been implicated in the occurrence of cavitation, resulting in the extraction of components that are released from cells during the drying process.

The antioxidant activity of many foods has been extensively studied due to its ability to counteract oxidation processes that reduce chronic illnesses associated with oxidative stress in the human body [48]. Various antioxidant chemicals, including ascorbic acid, flavonoids, and phenolic acids, were regarded as natural sources in horticulture goods. The ABTS analysis was employed to assess the antioxidant potential of both lipophilic and hydrophilic antioxidants, encompassing flavonoids (flavones, flavanones, and flavonols) and phenolic acids, particularly ferulic acid and p-coumaric acid [49]. The antioxidant capacity of fresh blood orange slices was determined to be 7380.01 mg TE/100g DM in the DPPH assay and 242.67 mg TE/100g DM in the ABTS assay. Consistent with the TPC results, the maximum DPPH and ABTS values recorded were 7330.09 mg TE/g DM and 226.46 mg TE/g DM, respectively, in FD-dried blood orange slices. At 50 °C, the UAVD technique yields 6907.63 mg TE/100g DM, followed by the FD method at 7339.09 mg TE/100g DM for DPPH, with the VD method resulting in 5990.63 mg TE/100g DM thereafter. A comparable pattern observed with DPPH is also evident with ABTS. Thermal treatments and oxidative processes may have caused the breakdown of phenolic compounds and reduced the antioxidant activity of the samples [50]. The antioxidant capacity of fruits dried using the UAVD technique surpasses that of samples dried using HAD, as indicated by TPC findings. The results indicate that the UAVD approach could be beneficial as an alternative to the HAD method.

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Table 4. Total phenolic content, antioxidant activity, and vitamin C values of fresh and dried blood orange.

	TPC (mg GAE/100g)		
Fresh	154.25±4.65^{A}	154.25±4.65^{A}	154.25±4.65^{A}
FD	131.27±0.41^{B}	131.27±0.41^{B}	131.27±0.41^{B}
	50°C	**60°C**	**70°C**
HAD	107.08±0.27^{Da}	87.99±0.41^{Db}	44.00±1.90^{Ec}
VD	122.24±1.08^{Ca}	112.36±1.49^{Cb}	59.70±0.0.27^{Dc}
UAVD	128.77±0.95^{Ba}	108.16±1.08^{Cb}	65.25±2.03^{Cc}
	DPPH (mg TE/100g)		
Fresh	7380.01±16.44^{A}	7380.01±16.44^{A}	7380.01±16.44^{A}
FD	7339.09±8.84^{A}	7339.09±8.84^{A}	7339.09±8.84^{A}
	50°C	**60°C**	**70°C**
HAD	5083.67±36.81^{Da}	4705.49±6.69^{Db}	2249.00±46.85^{Dc}
VD	5990.63±20.08^{Ca}	5123.83±36.81^{Cb}	4434.40±50.20^{Cc}
UAVD	6907.63±46.85^{Ba}	5950.476.69^{Bb}	4926.3753.55^{Bc}
	ABTS (mg TE/100g)		
Fresh	242.67±7.03^{A}	242.67±7.03^{A}	242.67±7.03^{A}
FD	225.46±0.35^{B}	225.46±0.35^{B}	225.46±0.35^{B}
	50°C	**60°C**	**70°C**
HAD	162.93±6.67^{Ea}	139.74±3.16^{Eb}	118.31±2.81^{Ec}
VD	185.76±0.70^{Da}	153.80±1.76^{Db}	129.56±1.41^{Dc}
UAVD	218.08±4.22^{Ca}	192.79±7.03^{Cb}	169.60±2.11^{Cc}
	Vitamin C (mg/100g)		
Fresh	55.32±0.45^{A}	55.32±0.45^{A}	55.32±0.45^{A}
FD	49.01±0.21^{B}	49.01±0.21^{B}	49.01±0.21^{B}
	50°C	**60°C**	**70°C**
HAD	36.19±0.14^{Da}	33.60±0.35^{Eb}	29.79±0.18^{Ec}
VD	44.81±0.30^{Ca}	41.40±0.25^{Db}	37.55±0.29^{Dc}
UAVD	46.10±0.08^{Ca}	44.82±0.44^{Ca}	41.63±0.20^{Cb}

*DR: Drying rate and **D_{eff}: Effective moisture diffusivity. Different lowercase letter displays significance of the parameters($p<0.5$). Different uppercase letter displays significance of the parameters($p<0.5$). .

3.3. Vitamin C

The concentrations of different chemical constituents, such as vitamins, minerals, and phenolics, which are recognized for their potent antioxidant qualities, impact the quality of citrus fruits [51]. Fresh juice has high levels of vitamin C, which is measured as ascorbic acid among the vitamins [52]. Rapisarda [53] reported that vitamin C is rich in blood oranges; in fact, Moro and Tarocco have a higher concentration of vitamin C than many other blood orange cultivars, with juice containing 0.50 to 0.80 g/kg. The vitamin C content of both fresh and dried blood orange slices is also shown in Table 4. Fresh blood orange slices have a vitamin C level of 55.32 mg/100g, whereas dried blood orange slices have a vitamin C content of 29.79 to 49.01 mg/100g, which is quite comparable to what has been found in other studies [46, 54].

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Table 7. Individual phenolic compounds of blood orange slices (µg/100mL of dry weight).

	FRESH	FD	HAD			VD			UAVD		
			50°C	60°C	70°C	50°C	60°C	70°C	50°C	60°C	70°C
4-Hydroxybenzoic acid	698.384	679.661	617.872	530.046	415.985	665.501	548.873	449.317	687.720	563.045	464.280
Gallic acid	694.133	663.086	586.187	506.159	386.882	608.642	563.000	420.385	630.301	585.626	458.424
Protocatechuic acid	90.007	76.274	50.086	34.536	29.728	60.823	52.608	51.665	81.743	87.068	59.782
Syringic acid	419.783	313.852	151.958	127.316	73.126	135.800	122.398	118.860	137.515	112.840	111.849
Caffeic acid	773.416	741.618	645.529	566.257	412.475	665.606	537.631	402.346	696.803	545.505	405.051
Chlorogenic acid	874.063	828.764	807.190	629.639	469.726	824.394	719.540	570.502	887.985	733.310	588.280
Ferulic acid	**2442.164**	**2307.669**	**1723.559**	**1586.942**	**1223.456**	**1927.890**	**1884.938**	**1645.212**	**2142.062**	**1922.387**	**1633.186**
o-coumaric acid	315.635	300.019	236.401	200.192	114.675	276.441	227.648	130.081	272.344	231.669	143.211
p-coumaric acid	902.509	910.730	835.561	726.757	622.768	853.823	773.700	651.752	876.964	757.289	683.279
Sinapic acid	569.698	547.795	439.647	387.185	265.075	459.391	354.217	295.903	498.377	339.098	259.853
Ellagic acid	1137.644	1075.601	814.108	756.400	529.044	911.640	844.927	708.886	980.226	860.551	731.712
Catechin	**1796.558**	**1407.770**	**1046.529**	**885.411**	**874.530**	**1264.428**	**1088.181**	**892.293**	**1279.250**	**1284.575**	**856.867**
Chrysin	698.898	684.894	604.188	400.100	241.552	630.269	568.110	451.798	670.510	550.835	452.889
Hesperidin	**12,201.82**	**11,766.77**	**8,445.41**	**6,978.38**	**5,811.42**	**9,832.24**	**9,036.76**	**7814,85**	**10,710.31**	**9,419.63**	**7,757.89**
Quercetin	265.783	219.296	182.930	146.665	127.057	152.290	125.304	108.828	178.698	134.023	106.376
Rutin	380.074	374.675	194.474	179.055	129.941	255.517	186.065	106.053	244.775	150.100	139.097

Table 8. Color parameters of blood orange slices.

	FRESH	FD	HAD			VD			UAVD		
			50 °C	60 °C	70 °C	50 °C	60 °C	70 °C	50 °C	60 °C	70 °C
L*	54.07±0.61^{a}	52.70±0.16^{a}	44.48±0.25^{d}	43.20±0.10^{d}	40.44±0.07^{f}	49.75±0.13^{b}	47.57±0.31^{c}	42.51±0.09^{de}	46.73±0.16^{c}	43.42±0.26^{d}	42.14±0.09^{de}
a*	18.35±0.14^{ab}	19.38±0.41^{a}	14.80±0.10^{d}	12.46±0.19^{e}	9.90±0.18^{f}	17.64±0.04^{b}	16.34±0.02^{c}	14.97±0.09^{d}	19.35±0.23^{a}	18.72±0.08^{ab}	17.57±0.31^{b}
b*	16.11±0.02^{a}	15.38±0.26^{ab}	12.17±0.15^{c}	9.98±0.54^{d}	5.11±0.12^{f}	14.34±0.28^{b}	12.26±0.15^{c}	6.74±0.20^{ef}	14.14±0.15^{ab}	12.71±0.18^{c}	7.51±0.12^{e}

Different lowercase letter displays significance of the parameters ($p<0.5$). Different uppercase letter displays significance of the parameters($p<0.5$). .

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3.4. Individual phenolic compounds

Table 7 shows the effect of different drying methods and temperatures on individual phenolic compounds of blood orange slices. The HAD method caused greater reductions in the amount of all phenolic compounds identified than other drying methods. Hirsch [55] indicated that the activation of oxidative enzymes, including polyphenol oxidase, during hot air oven drying results in a reduction of flavonoid concentration. The loss was reduced due to the diminished activity of the polyphenol oxidase enzyme during freeze-drying at lower temperatures [56].

There are 6 hydroxycinnamic acids found in blood orange slices which are caffeic acid, chlorogenic acid, ferulic acid, o-coumaric acid, p-coumaric acid, and sinapic acid. Ferulic acid was the most dominant hydroxycinnamic acid in fresh and dried blood orange slices, accounting for the largest proportion of the total hydroxycinnamic acid contents. Ferulic acid is found mostly in fresh blood oranges (2442.164 mg/100gDM). Among dried blood oranges, freeze-dried blood oranges contained the most ferulic acid (2307.669 mg/100 gKM), followed by blood oranges dried with UAVD at 50 °C with a ferulic acid content of 2142.062 mg/100 gKM. Ellagic acid is a hexahydroxydiphenic acid abundantly found in fruits, pomegranates, cranberries, and other plant foods. This study found that it is the most abundant compound in blood orange after ferulic acid. Ellagic acid exhibits a wide range of biological properties, such as playing an active role in anti-cancer treatment [57].

Flavonoids isolated from citrus fruits are a group of natural compounds with phenolic structures. This study found 6 flavonoids chrysin and rutin were flavon, catechin, myricetin and quercetin were flavanol, and hesperidin was flavanone. Flavanone is the major flavonoid in orange varieties. Table 7 shows that hesperidin was a flavanone and the most abundant individual phenolic compound in fresh and dried blood orange slices. Hesperidin is found mostly in fresh blood oranges (12,201.82 mg/100gDM), followed by freeze-dried blood oranges (11,766.77 mg/100gDM).

3.5. Color

The critical factor of dried products influencing consumer acceptance is product color. Table 8 displays the color characteristics of fresh and dried blood orange slices. The L*, a*, and b* values of fresh blood orange slices are 54.07, 18.35, and 16.15, respectively. In contrast, the L* values of dried blood orange slices vary from 40.44 to 52.70, the a* values range from 9.90 to 19.38, and the b* values range from 5.11 to 16.15. The L*, a*, and b* values of the FD dry blood orange slices were the highest among all dried blood orange slices and were closer to the L*, a*, and b* values of fresh blood orange slices. The L*, a*, and b* values of blood orange slices dried using the freeze-drying method are then compared to those dried using the UAVD method at 50 °C. According to certain researches [10, 11, 37], the UAVD approach can avoid color changes throughout the drying process. Turan et al. [37] indicated elevated L*, a*, and b* values for UAVD-dried goji berries in comparison to HAD-dried samples. The reduction in the L* value of dried samples is mostly attributable to Maillard reactions and nonenzymatic browning, which are inevitable during drying operations [58]. Furthermore, drying may diminish essential substrates for the Maillard reaction, including sugars and soluble pigments, resulting in a decrease in the a* value of the dried samples [59].

4. Conclusions

This study investigated the effects of different drying methods and temperatures on drying duration, bioactive compounds, vitamin C levels, and color alterations in blood oranges. UAVD demonstrated decreased drying time and improved retention of bioactive constituents, such as vitamin C, in comparison to VD and HAD. Moreover, UAVD exhibited less color changes and shrinkage relative to VD and HAD. Despite the samples dried by freeze-drying (FD) demonstrating the highest retention of bioactive substances, their total phenolic content (TPC) and CUPRAC recovery values were lower than those of the other samples. This study demonstrates that UAVD is

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efficacious for drying raspberry fruit, leading to decreased drying duration, negligible color and physical changes in samples, and improved retention of bioactive components.

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Abbreviations

The following abbreviations are used in this manuscript:

ABTS	2,2'-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid
D_{eff}	Effective moisture diffusivity
DPPH	2,2-diphenyl-1-picrylhydrazyl
DR	drying rate
FD	Freeze-drying
HAD	Hot air drying
HPLC	high-performance liquid chromatography
M_e	Equilibrium moisture content
M_t	Moisture content at time t
M_0	Initial moisture content
MR	Moisture ratio
MR_{prei}	Predicted moisture ratio
MR_{expi}	Experimentally determined moisture ratio
RMSE	Root mean square error
TPC	Total phenolic content
UAVD	Ultrasound-assisted vacuum drying
VD	Vacuum drying

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