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HOT-AIR DRYING AND DEGRADATION KINETICS OF BIOACTIVE COMPOUNDS OF GILABURU (*Viburnum opulus* L.) FRUIT

Article Highlights

- Thermal degradation of selected bioactive compounds were fitted to the first-order kinetic model
- The drying rate of gilaburu fruit was highly influenced by drying temperature
- The effective diffusion coefficient increased with increasing drying temperature
- Selected bioactive compounds of gilaburu were reduced by the drying process
- The Parabolic and Page models were determined to predict the experimental drying best

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Abstract

This study aims to determine whether drying is a suitable preservation method for gilaburu fruit and the changes in the bioactive components of gilaburu fruit (Viburnum opulus L.) at the end of the drying process. In this study, gilaburu fruits were dried in a cabinet dryer at different temperatures (50 °C, 60 °C, and 70 °C). The analyses of trans-resveratrol, water-soluble vitamins, organic acids, and phenolic compounds were made using the HPLC method, while total phenolic contents and antioxidant activity were spectrophotometric. As a result of drying of gilaburu fruit at 50 °C, 60 °C, and 70 °C, the highest component loss was observed at 70 °C. Losses of 73.64% and 84.08%, respectively, were detected in the total phenolic substance and antioxidant capacity content of gilaburu fruit after drying at 70 °C. While the trans-resveratrol content was 1.26±0.05 (g/100 g dry weight (DW)) in fresh fruit, it reduced to 0.31±0.03, 0.30±0.01 and 0.21±0.01 after drying at 50 °C, 60 °C and 70 °C, respectively. In terms of vitamins, the highest loss was seen in niacin. The contents of ascorbic acid, pyridoxine, niacin and thiamine contents of fresh gilaburu fruit decreased after drying at 50 °C, 60 °C and 70 °C. In addition, drying kinetics of water-soluble vitamins, total phenolic contents, antioxidant activity, and trans-resveratrol were modeled. The Page model best described the drying behavior of fruits at 70 °C, and the parabolic model at both 50 °C and 60 °C. Thermal degradation of water-soluble vitamins, total phenolic contents, antioxidant activity, and trans-resveratrol were fitted in the first-order kinetic model.

Keywords: antioxidant capacity, drying kinetic, gilaburu, trans-resveratrol, total phenolic content, water-soluble vitamins.

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Fruits contain many nutritive and non-nutritive bioactive components such as flavonoids, phenolic acids, tannins, carotenoids, vitamins, sugars, minerals, and essential oils [1]. Edible wild fruits have played an important role in nutrition with their rich biodiversity since the beginning of humanity [2]. Gilaburu (*Viburnum opulus*) is one of the wild fruits originating from North Africa, North Asia, and Europe grown mainly in the Central Anatolia Region in Turkey and does not require high climatic features and can be grown in

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almost every region where there is water [3]. Gilaburu plant (*Viburnum opulus* L.), which is from the Caprifoliaceae (Honeysuckle) family of the Dipsacales (Rubiales) team, has more than 230 species, most of which are endemic [4]. Four different species of Viburnum (*Viburnum tinus* L., *Viburnum lantana* L., *Viburnum orientale* P., and *Viburnum opulus* L.) are grown in Turkey [5]. While it is known by various names such as European cranberrybush, American cranberry bush, and cranberry tree worldwide, it is known as gilaburu in Turkey [6].

The ripening of the gilaburu fruit is completed in September-October, and clusters with 30–40 fruits are formed. The gilaburu plant's ripe fruits are bright red, round-oval in shape, single-seeded, thin-shelled, and juicy. Ripe fruits are acidic and bitter [7,8]. Gilaburu fruits can be consumed as they are plucked from the branch. However, since it has a bitter and acrid taste, it is preferred to be consumed in brine or fruit juice by adding sugar and water [9–11].

In recent years, the use of isolated plants has become popular in the world [12]. The gilaburu plant, which has been cultivated since the "16th century", has also been used in the treatment of many diseases such as stomach pain, gall bladder disorders, kidney stones, liver diseases, diuretics, menstrual pain, prevention of miscarriage and bleeding, mumps, diabetes, hemorrhoids using the fruit, leaves, and shells [13–15]. Gilaburu fruit is a good source of vitamin C. Also, it contains vitamins A and E, micronutrients (Cu, Mn, Fe, and Zn) and macronutrients (P, K, Mg, Ca, and N), organic acids, fatty acids, and phenolic compounds [16–20]. Phenolic compounds act as natural antioxidants which protect the plant from external factors. *Trans*-resveratrol, a phenolic acid in a *trans*-isomer structure, has many health benefits since it is an anticarcinogen, anti-inflammatory, antioxidant, heart protective, and vasodilator [21–23].

Drying is one of the most common preservation methods for fruits and vegetables [24]. Commonly used drying methods are natural drying in the sun and industrial drying in tray cabinet dryers. Although sun drying is a process that requires low cost, it has some disadvantages [25]. Although drying with cabinet dryers is more costly than sun drying, there is minimal loss of nutritional value and better physical preservation thanks to adjustable time and temperature parameters, a fast drying process, and homogeneous drying [26,27]. Almost half of the worldwide dried fruit market consists of raisins, followed by figs, apricots, peaches, and apples [28]. Gilaburu is a fruit that is generally used after brining. It is important to investigate the potential of dried gilaburu fruit. In the literature, studies investigate the pH, titration acidity, total phenolic content, antioxidant capacity, color, and texture values of the fruit after drying [29–31]. However, there is a dearth of information on the water-soluble vitamins, organic acids, *trans*-resveratrol, and phenolic compounds of dried gilaburu fruit. The current study presents the importance of changes in vitamin, organic acid, total phenolic content, and antioxidant capacity of gilaburu fruit due to drying with hot air. In addition, there is no data on the *trans*-resveratrol content of gilaburu fruit in previous scientific studies.

This study aims to determine the effect of drying on selected biochemical compounds and kinetic characteristics of gilaburu fruit for industrial purposes. In addition, determining the drying characteristics and creating mathematical models to determine the most suitable drying parameters at different temperatures are targeted.

MATERIAL AND METHODS

Sample collection

In this study, *Viburnum opulus* L. species of gilaburu fruit was used as material. The samples were obtained from Kayseri province (Kayseri Pazarı Bio Herbal Products Limited Company). Gilaburu fruits were collected homogeneously from 10 randomly selected plants in a private garden and brought to the laboratory by a refrigerated vehicle. The ripe fruits used in the analysis were selected. Fresh fruits were stored at -18 °C until analysis [32].

Drying process

Gilaburu fruits were dried in a drying cabinet (Yücebaş Makine Tic. LTD. ŞTİ. İzmir, Turkey) until the moisture content of samples reached up to 18%–20% on a wet basis. The tray cabinet dryer consisted of a resistance heater providing the temperature, a temperature control panel, and a fan providing the airflow (EUC442 model, ENDA, Turkey). The cabin, which has dimensions of 70 cm x 55 cm x 100 cm, operates in the temperature range of 40 °C–120 °C, air flow rate of 2 m/s, and relative humidity of 20%–95%. The drying process was carried out at three different temperatures, (50, 60, and 70) °C. The drying process was applied three times, including a preliminary trial drying for all three temperature values. Before drying, the cabinet was preheated until it reached the specified drying temperature. Drying tests were carried out with 200 g samples to determine the time norms of the temperature parameters. The samples (2000 g) were distributed homogeneously on the drying trays (25 cm x 20 cm x 3 cm). Average air velocity and relative humidity of 2 m/s and 20% were recorded, respectively. Gilaburu fruits were spread homoge-

neously as a single layer on the drying tray. During the drying process, fruits were weighed every half hour for the first 5 hours and then at one-hour intervals for the additional hours. The drying rate was calculated by recording these data.

Drying characteristics of gilaburu fruit

Knowing the moisture content is an important parameter for calculating mathematical models. Eq. (1) was used to calculate the humidity ratio:

$$MR = \frac{M_t - M_e}{M_i - M_e} \quad (1)$$

where MR is the moisture ratio of samples (dimensional), M_i and M_t are the initial and actual (at time t) moisture content of the sample (g water g^{-1} DW), respectively, and M_e is the equilibrium moisture content of example at t time (g water g^{-1} DW).

To determine the moisture content in the food drying process, M_t, M_i, and M_e values are compared. Since the M_e value is very low compared to the others, it is accepted as 0 in the calculations, and the humidity ratio is calculated using Eq. (2) [33]:

$$MR = \frac{M_t}{M_i} \quad (2)$$

The drying rate is determined by using Eq. (3).

$$Drying\ rate = \frac{M_{t+\Delta t} - M_t}{\Delta t} \quad (3)$$

where M_t is the moisture content of the sample for any time (g water g^{-1} DW), $M_{t+\Delta t}$ is the moisture content of the sample at any t+Δt time (g water g^{-1} DW), and Δt is the time difference between two measurements (hours).

Mathematical models examine the effects of ambient conditions such as air temperature, humidity, and flow rate [34]. The coefficient of determination (R^2), estimated standard error (RMSE), and chi-square ($\chi 2$) values are used when explaining the relationship between the estimated and experimental data of the samples dried at different temperatures. The model with the highest R^2 value and the lowest $\chi 2$ and RMSE should be selected to determine the best model for explaining the relationship between experimental and predicted data. MATLAB (R2015a) program was used to calculate the mathematical modeling data. The mathematical models used in this study are given in Table 1.

The RMSE and the chi-square ($\chi 2$) value were calculated by using Eqs (4) and (5), respectively.

$$RMSE = \left[\frac{1}{N}\sum_{i=0}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2\right]^{1/2} \quad (4)$$

$$\chi^2 = \frac{\sum_{i=0}^{N}\left(MR_{pre,i} - MR_{\exp,i}\right)^2}{N - n} \quad (5)$$

where $MR_{pre,i}$ and $MR_{exp,i}$ are the predicted and experimental moisture ratios, respectively, N is the number of experimental data, and n is the constants of thin layer drying models.

Table 1. Mathematical models.

Model name	Model	References
Parabolic	$a + bt + ct^2$	[33]
Logarithmic	$aexp(-kt) + c$	[35]
Lewis	$exp(-kt)$	[36]
Henderson and Pabis	$aexp(-kt)$	[37]
Page	$exp(-kt^n)$	[37]
Wang and Sing	$1 + at + bt^2$	[38]

Calculation of effective moisture diffusion and activation energy in hot air drying

The drying process is based on the principle that water molecules move from the place where the density of the molecules is more to the place where it is less. This situation is explained by Fick's law of diffusion [39]. Crank [40] proposed Eq. (6) to calculate the effective moisture diffusion in spherical products, provided there is no shrinkage in the dried material, and the effective diffusion is constant [41]:

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

where D_{eff} is the effective moisture diffusivity (m^2 s^{-1}), and r is the arithmetical average of the radius of samples at measured intervals (m).

Eq. (6) was shortened to Eq. (7) [37]:

$$\ln(MR) = \ln\left(\frac{6}{\pi^2}\right) - \left(\frac{\pi^2 D_{eff}t}{r^2}\right) \quad (7)$$

The slope of the graph drawn by Eq. (7) was calculated by Eq. (8):

$$Slope = -\frac{\pi^2 D_{eff}}{r^2} \quad (8)$$

The Arrhenius equation was used to calculate the activation energy in the hot air drying process [42]:

$$D_{eff} = D_0\exp\left(\frac{-E_a}{RT}\right) \quad (9)$$

where R is the universal gas constant (8.314 J mol^{-1} K^{-1} or 1.987 cal mol^{-1} K^{-1}, T is the actual temperature (K), E_a is the activation energy (kJ mol^{-1} or kcal mol^{-1}), and D_0 is the constant before exponential (m^2 s^{-1}).

Eq. (10) is obtained using the natural logarithm of

Eqs. (8) and (9):

$$\ln D_{eff} = \ln D_0 - \frac{E_a}{RT} \quad (10)$$

The slope of the graph plotted against T^{-1} of the natural logarithm of the effective diffusion coefficient gives the activation energy.

Analysis of *trans*-resveratrol

Trans-resveratrol, a phenolic component, was analyzed according to the method suggested by Singh and Pai [43]. Methanol was used to extract gilaburu fruits, which are dried at different temperatures. An HPLC device (SHIMADZU LC20AD) consists of a column oven (SHIMADZU CTO-20A), column (ACE C18 (7.8 mm x 300 mm)), a pump (SHIMADZU LC-20AD), a degasser (SHIMADZU DGU-20A3), and a photodiode array (PDA) detector (SPDM20A), was used for *trans*-resveratrol analysis.

The calibration curve of the *trans*-resveratrol standard was prepared at (5, 10, 25, 50, 75, and 100) mg/L concentrations. Calculations were made using the equation (y=177599x-308529) of the calibration curve with a high R^2 (0.9985) value drawn with these concentrations. The method used for *trans*-resveratrol analysis is given in Table 2.

Analysis of water-soluble vitamins

An HPLC device (SHIMADZU LC20AD) was used to analyze water-soluble vitamins. The water-soluble vitamin analysis was carried out by modifying the method suggested by Otağ [19]. The method used in the analysis is given in Table 2. The water-soluble vitamin content was calculated using the equation obtained from the calibration curve with a high R^2 value using the stock solutions prepared at different concentrations (5, 10, 25, 50, 75, and 100) mg/L. The R^2 values obtained for ascorbic acid, pyridoxine, niacin, and thiamine were found to be 0.9984 (y=83790x-432582), 0.9997 (y=36871x+11924), 0.9999 (y=30299x+11105), and 0.9993 (y=62655x+128944), respectively.

Analysis of organic acids

Organic acid analysis was carried out by modifying the method proposed by Soyer et al. [44]. The method of this analysis performed with an HPLC device (SHIMADZU LC20AD) is given in Table 2. Standard calibration curves of organic acids were created with standards prepared at 100, 250, 500, 750, and 1000 mg/L concentrations. The R^2 values of the calibration curves were found to be 0.9998 (y=1520.9x+3100.3), 0.9999 (y=1065.9x-1974), and 0.9998 (y=857.83x-40.273) for tartaric, citric and malic acid, respectively. Calculations were made with the equation obtained from this calibration curve.

Analysis of phenolic compounds

Methanol extraction of the phenolic component composition of gilaburu samples dried at different temperature parameters was carried out, modifying the method suggested by Choi *et al.* [45]. The modification of the method suggested by Gao *et al.* [46] was finally used to extract phenolic compounds. Phenolic compounds were identified by modifying the method proposed by Bansal *et al.* [47]. Two different mobile phases (gradient) were used in this method. The operating conditions of the HPLC device (SHIMADZU LC20AD) used to detect phenolic compounds are given in Table 2. Standard calibration curves of phenolic compounds were prepared at (5, 10, 25, 50, and 100) mg/L concentrations. Calculations were made using the calibration curve equation with a high R^2 value. The highest R^2 values detected for chlorogenic, ellagic, p-coumaric, caffeic acid, and rutin were 0.9998 (y=64035x-63331), 0.9998 (y=178344x-306186), 0.9999 (y=283357x-230476), 1 (y=120497x-40235), and 1 (y=61226x-26563), respectively.

Total phenolic content and antioxidant activity analysis

The total phenolic content (TPC) of gilaburu samples, which were dried at different temperature parameters, was determined spectrophotometrically by modifying the method suggested by Singleton and Rossi [48]. The gallic acid curve created for the calculations was prepared using (25, 50, 75, and 100) mg/L standards. Calculations were made using the equation of the calibration curve (y=0.0097x+0.0834; R^2=0.9977) drawn with these concentrations. The absorbances of the samples were read in a spectrophotometer (PG Instruments T80 UV/VIS, UK) at a wavelength of 760 nm. Analysis results are given as mg gallic acid equivalent (GAE)/100 g DW.

The extracts used in the total antioxidant activity (AC) analysis of gilaburu samples were prepared using the same method as the methanol extracts prepared to determine phenolic compounds. Analysis was performed spectrophotometrically by the method of DPPH (2.2 diphenyl-1-picrylhydrazyl) proposed by Thaipong et al. [49]. The absorbances of the samples and standards were read in a spectrophotometer (PG Instruments T80 UV/VIS, UK) at a wavelength of 515 nm. The results were calculated in mmol Trolox equivalent (mmol TE)/g DW according to the equation obtained by preparing the standard curve (y=-0.017x+1.0278; R^2 =0.9853) of Trolox (Sigma-Aldrich Chemie gmbh) at (10, 20, 25, 30, and 50) mg/L.

Table 2. Methods used in chromatographic analyses.

		Column	Flow rate	Oven temperature	Wavelength	Mobile phase
Water-Soluble Vitamins	Ascorbic acid	ACE C18	0.8 ml/min	40°C	254 nm	0.1 M KH_2P0_4+0.1 M KOH
	Pyridoxine				324 nm	
	Niacin				261 nm	
	Thiamin				234 nm	
Organic Acids	Tartaric Acid		1.0 ml/min	25°C	214 nm	0.01 N H_2SO_4
	Citric Acid					
	Malic Acid					
Phenolic Compounds	Chlorogenic acid		0.5 ml/min		280 nm	0.1 orto-H_3PO_4:C_2H_3N
	Ellagic acid				254 nm	
	p-Coumaric acid				280 nm	
	Caffeic acid					
	Rutin				360 nm	
Trans-resveratrol			0.8 ml/min	30°C	306 nm	Metanol:10mM KH_2PO_4: C_2H_3N

Statistical analysis

SPSS software statistical package program (SPSS ver. 23, SPSS Inc., Chicago, IL, USA) was used to analyze the data. One-way analysis of variance (ANOVA) was used to evaluate differences between treatments with a significance level of $p<0.05$. Duncan's multiple comparison test was used to determine the difference between groups. All analyses were carried out in duplicate.

RESULTS AND DISCUSSION

Drying characteristic of whole gilaburu fruits during hot air drying

The moisture ratio (A) and drying rate (B) of gilaburu fruits during hot air drying are shown in Figure 1. Initially, the moisture content of gilaburu fruit was determined as 83.95%. A decrease in moisture content was observed over time during drying with hot air. An increase was observed depending on the drying rate of the gilaburu fruits with hot air. Accordingly, the drying time was reduced to 75 (4500 min), 17 (1020 min), and 7 h (420 min) for (50, 60, and 70)°C, respectively (air velocity 2 m s^{-1}). In a study conducted with gilaburu samples obtained from the Kayseri region, the samples were dried at an air velocity of 1.3 m/s. The (60, 70 and 80) °C drying process was completed in (2663, 856, and 420) minutes, respectively [29]. Considering the air velocity, it was observed that the drying times were similar. Heat transfer is provided by increasing the temperature difference [50]. As the temperature difference increases, more energy is transferred to fresh gilaburu fruits, and thus more water evaporates from the content of gilaburu fruits per unit of time. In addition, the increase in temperature decreased the relative humidity of the drying air, so the water transfer from the structure of gilaburu fruits to the drying air accelerated. In this case, the shortening of the drying time due to the increase in temperature can be explained by the increase in mass transfer [51].

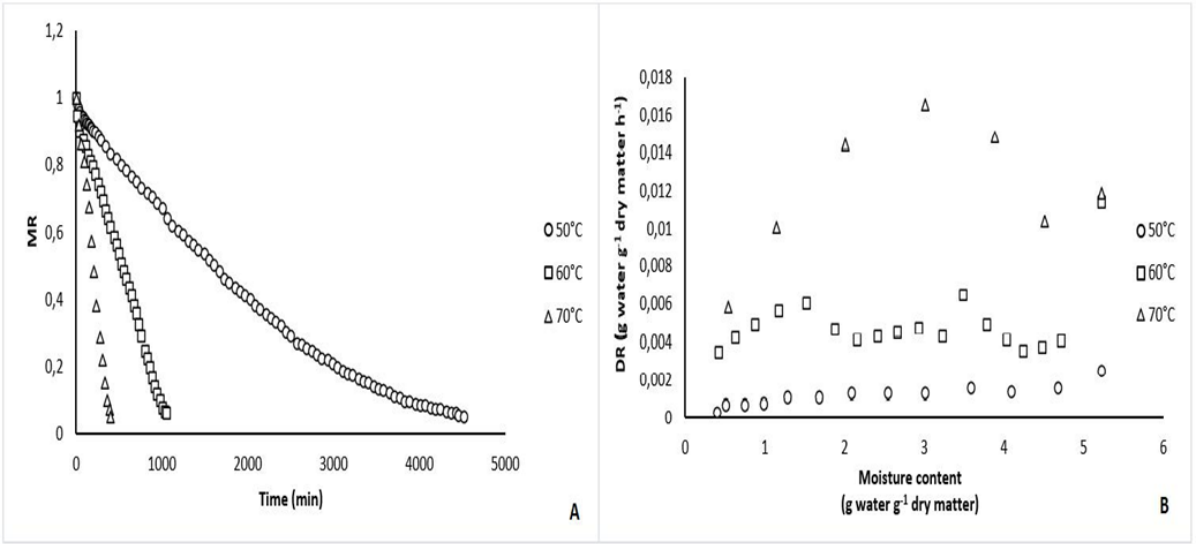

Figure 1. Moisture ratio (MR) and drying rate (DR) of whole gilaburu fruits during hot air drying.

The moisture ratio of gilaburu fruits during hot air drying was used to be fitted with mathematical models. The models used are listed in Table 1. The most suitable mathematical model giving the lowest RMSE and χ^2 and the highest R^2 value was preferred [37]. The parabolic model best describes the experimental MR of gilaburu fruits dried at 50 and 60 °C. In addition, the Page model was the best model to describe the experimental MR of fruits dried at 70 °C (Table 3).

Effective moisture diffusivity and activation energy of whole gilaburu fruits during hot air drying

Table 4 presents the D_{eff} and E_a values of gilaburu fruits. It was observed that the effective diffusion coefficient increased in accordance with the temperature increase [52]. The effective diffusion coefficient is a positive indicator of dehydration efficiency. A high D_{eff} value indicates a fast drying process [53]. The increase in the D_{eff} value because of the increase in temperature indicates that the moisture will be removed from the gilaburu fruit more easily.

In the literature, the D_{eff} and E_a values of drying of gilaburu fruits have not been found; however, there are similar studies. As a result of the calculations, the E_a value was determined to be 133.81 kJ mol^{-1}. In a study conducted with goji berry fruit, similar to gilaburu fruit, the E_a value was 48.37 kJ mol^{-1} [42]. It is thought that

Table 3. Thin-layer mathematical models, models constants, and statistical parameters of thin-layer drying curves.

Models	Temperature	Model constants			χ^2	RMSE	R^2
Parabolic	50°C	a= 0.977	b= -0.0003537	c= 0.00000003274	0.000044974	0.0066	0.9996
	60°C	a= 0.9589	b= -0.0008386	c= -0.00000003899	0.00019453	0.0133	0.9979
	70°C	a= 1.025	b= -0.0027	c= 0.000000478	0.001380291	0.0332	0.9915
Logarithmic	50°C	k= 0.0005596	a= 0.9708	c= 0.0529	0.002201172	0.0461	0.9791
	60°C	k= 0.001814	a= 0.9984	c= 0.0606	0.006625917	0.0780	0.927
	70°C	k= 0.005076	a= 1.069	c= 0.051	0.012675613	0.1007	0.9151
Lewis	50°C	k= 0.0004904			0.001366086	0.0367	0.9865
	60°C	k= 0.001529			0.005459036	0.0728	0.9345
	70°C	k= 0.004131			0.010951296	0.1011	0.9078
Henderson	50°C	k= 0.0004996	a= 1.016		0.001341656	0.0362	0.9871
and Pabis	60°C	k= 0.001628	a= 1.053		0.005281129	0.0706	0.9401
	70°C	k= 0.004654	a= 1.116		0.009891817	0.0926	0.9283
Page	50°C	k= 0.0001023	n= 1.204		0.000527738	0.0227	0.9949
	60°C	k= 0.0000786	n= 1.463		0.002212666	0.0457	0.9749
	70°C	k= 0.00004651	n= 1.821		0.001070552	0.0304	0.9922
Wang and Singh	50°C	a= -0.0003746	b= 0.00000003662		0.00015759	0.0124	0.9985
	60°C	a= -0.0009923	b= 0.00000008021		0.000439509	0.0204	0.995
	70°C	a= -0.002465	b= 0.00000001802		0.001294131	0.0335	0.9906

Table 4. Effective moisture diffusivity and activation energy of gilaburu.

Temperature	D_{eff} (m^{2}s^{-1})	E_a (kJ mol^{-1})
50°C	1.82x10^{-11}	
60°C	4.01x10^{-11}	133.814
70°C	3.38x10^{-10}	

the differences between E_a values might be due to different drying conditions and fruit types. D_{eff} values obtained from drying gilaburu fruit increased depending on the temperature increase. In a study with grapes, the drying process was examined at (30, 35, 40, and 45) °C, and the highest D_{eff} value was at 45 °C, the highest drying temperature [54]. It was seen that the analyzed data supported the present study. The Arrhenius equation correlating D_{eff} and T^{-1} is presented in Fig. 2.

The effect of the drying process

Changes in trans-resveratrol contents

In this study, the *trans*-resveratrol content of fresh fruits was determined to be 1.26 g/100 g. *Trans*-resveratrol content decreased due to the drying of gilaburu fruit with hot air at (50, 60, and 70) °C. The highest loss of *trans*-resveratrol content of gilaburu fruits was 82.90% at 70 °C, while the lowest loss was 75.02% at 50 °C. There is no study on the *trans*-resveratrol content of gilaburu fruit in the literature. However, in a study using blueberry juice, a spray drying process was applied, and the effect of the drying process on *trans*-resveratrol was evaluated. In this study, an average of 96% loss was observed in the *trans*-resveratrol content of the samples [55]. When the results were compared, it was observed that the temperature application caused a decrease in *trans*-

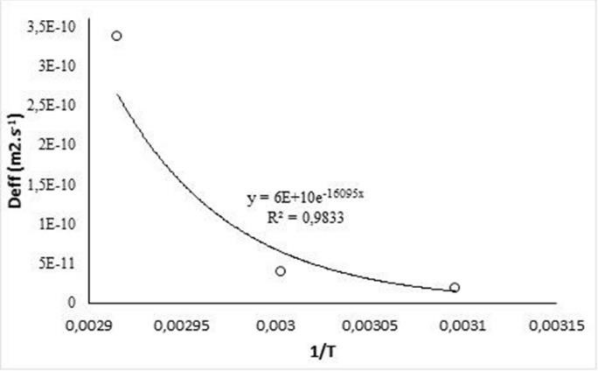

Figure 2. Arrhenius-type equation between D_{eff} and T^{-1}.

resveratrol. This decrease is because *trans*-resveratrol is a lipophilic polyphenol sensitive to thermal degradation [56].

Changes in water-soluble vitamin contents

Preserving heat-sensitive vitamins during drying is considered an indicator of food quality [57]. Especially ascorbic acid is a critical quality parameter. In general, if the loss of ascorbic acid is low after the applied process, it is thought that the loss of other nutritional elements is also low [58]. The change in water-soluble vitamin contents of gilaburu fruits at different drying temperatures is shown in Table 5. Ascorbic acid (vitamin C), thiamine (B1), niacin (B3), and pyridoxine (B6) analyses were performed in gilaburu fruits and the dominant vitamin was found to be ascorbic acid. In a study, it was determined that gilaburu fruit contains high levels of ascorbic acid [59]. A significant decrease in vitamin content was observed in parallel with the increase in drying temperature.

The ascorbic acid content of fresh gilaburu fruit was determined to be 0.78 g/100 g DW. In a study by Akbulut *et al.*, the ascorbic acid content of fresh gilaburu fruit was 0.59 g/100 g DW [60]. The ascorbic acid content of gilaburu fruits dried at (50, 60, and 70) °C were found to be (0.35, 0.30, and 0.24) g/100 g DW, respectively. In a study conducted with jujube fruits, it was reported that there was a decrease in the amount of ascorbic acid depending on the temperature increase [61]. This decrease in the ascorbic acid content of fruits is due to the low thermal sensitivity of ascorbic acid [56]. In the current study, the pyridoxine content of fresh gilaburu fruits was determined to be 3.14 mg/100 g DW. After drying at 70 °C, the pyridoxine content was decreased to 0.75 mg/100 g DW. The thiamine content decreased from 0.30 mg/100 g DW to 0.14 mg/100 g DW after drying at 70 °C. Heat treatment applied at high temperatures easily breaks the molecular ring structures and methylene group chemical bonds of thiamine, causing devitaminization [62]. The lowest amount of niacin was found in fresh fruits (0.12 mg/100 g DW). As a result of drying at 70 °C, niacin could not be detected. Niacin is a heat-stable compound due to the pyrimidine ring in its structure. However, due to the low initial niacin content of gilaburu fruit and the long drying time, niacin could not be detected at the end of drying [62]. In the literature, no study has been found investigating the vitamin B content of gilaburu fruit. These types of vitamins were found in a similar study with caper fruit [63]. In addition, in the study conducted by Duman with rosehip fruits, it was reported that there was a decrease in the content of thiamine and riboflavin after drying the rosehip fruit [64]. In a study, there was a decrease in vitamin values due to drying the jujube fruit at different temperatures [65].

Changes in organic acids contents

The dominant organic acid of gilaburu fruit was tartaric acid. In addition, the fresh fruit contains malic and citric acids. A study examining the organic acid content of gilaburu fruit reported that the dominant organic acid was tartaric acid [1]. Tartaric, citric, and malic acid contents of fresh gilaburu fruit were 11.06±0.23, 6.74±0.37, and 8.62±0.05, respectively. In a study conducted with 11 different gilaburu samples grown in different parts of the country, the amount of malic acid was between (578.0 and 2090.0) mg/100 g [66]. The same study reported that the amount of citric acid was in the range of (270.0–1630.0) mg/100 g. In another study, the dominant organic acid of fresh fruit was malic acid. In addition, tartaric, citric, and malic acid contents were reported to be 0.37±0.02, 3.09±0.01, and 3.13±0.02, respectively [67]. The results recorded in this study and the literature samples differ due to the factors, such as species diversity, climatic conditions, and soil properties, affecting the composition.

The changes in organic acid content after drying gilaburu fruits at different temperatures are shown in Table 5. While the tartaric acid value in fresh fruit was 11.06 g/100 g DW, after drying at (50, 60, and 70) °C, this value was (10.67, 10.54, and 10.35) g/100 g DW, respectively. While the malic acid value was 8.62 g/100 g DW in fresh fruit, it decreased to 8.11 g/100 g DW as the result of drying at 70 °C. Similarly, while the citric acid value in fresh fruit was 6.74 g/100 g DW, it decreased to 6.02 g/100 g DW after drying at 70 °C.

Generally, decreases in the organic acid values were observed with the increase in drying temperature. Adiletta *et al.* stated that the organic acid amount of red and white grapes decreased after drying at 50 °C [68] due to the decrease in organic acids and oxidation reactions [69].

Changes in phenolic components

The phenolic components of gilaburu fruit are shown in Table 5. Chlorogenic acid, ellagic acid, p-coumaric acid, caffeic acid and rutin analyses were carried out in gilaburu fruit. However, chlorogenic acid could not be detected. The dominant phenolic component of fresh fruits was caffeic and ellagic acid. After drying at all temperatures, the amount of phenolic compounds decreased. While the value of caffeic acid, the dominant phenolic component, was 0.64 g/100 g DW in fresh fruits, it decreased to 0.41 g/100 g DW after drying at 70 °C. Ellagic acid, the other dominant phenolic acid, decreased to 0.25 g/100 g DW after drying at 70 °C.

In a study conducted with fresh fruit, the chlorogenic, caffeic, and *p*-coumaric acid contents were in the ranges of (23.64–30.33, 14.82–19.92, and 6.38–11.18) mg/100 mL, respectively [70]. In another study, the flower, bark, and fruit of gilaburu were examined, and chlorogenic acid (752.59±2.07 mg/100 g) and rutin (5.39±0.03 mg/100 g) were detected in the fruit, but *p*-coumaric acid could not be detected [67]. In the literature, no study has been found on the changes in the phenolic composition of gilaburu fruit due to drying, but there are studies conducted with different fruits. In a study investigating the effect of drying with hot air on phenolic compounds, orange peel and pulp were used as materials. As a result, long-term heat treatment at high temperatures destroyed the phenolic compounds [71] because phenolic compounds have an easily oxidized structure [72].

Changes in total phenolic content and antioxidant capacity

The total phenolic content and antioxidant capacity values of gilaburu fruit are shown in Table 5. The increase in drying temperature decreased the total phenolic content of gilaburu fruits. While total phenolic content was 568.96 mg GAE/100 g DW in fresh fruit, that value decreased to 149.89 mg GAE/100 g DW after drying at 70 °C. A significant decrease was observed in the total phenolic content of gilaburu fruits dried at three different temperatures, depending on the drying process. The decrease in antioxidant activity due to the drying process is the loss of antioxidant compounds during drying [73]. Zarifikhosroshahi [17] determined the TPC value as 1009.89 mg GAE/100 g DW in gilaburu samples from the Kayseri region. This value was higher than the value obtained as a result of the present study. In another study conducted with gilaburu fruits obtained from the Kayseri region, the TPC value was determined as 633.56 mg GAE/100 g DW [74], which is close to the result of the present study. The antioxidant capacity of fresh fruit was detected at 15.08 µmol TE/g DW, and it decreased to 2.40 µmol TE/g DW after the drying process at 70 °C.

Table 5. Changes in the composition of gilaburu fruit after drying.

Analysis		Fresh	50 °C	Reduction %	60 °C	Reduction %	70 °C	Reduction %
Total phenolic content (mg GAE/100g DW)		568.97±21.33^{a}	351.46±6.18^{b}	38.22	233.80±7.52^{c}	58.90	149.96±4.87^{d}	73.64
Antioxidant capacity (mmol TE/g DW)		15.08±0.001^{a}	2.81±0.001^{b}	81.36	2.51±0.001^{d}	83.35	2.40±0.001^{c}	84.08
Ascorbic acid (g/100 g DW)	Ascorbic acid	0.78±0.32^{a}	0.35±0.04^{b}	55.12	0.30±0.01^{b}	61.53	0.24±0.07^{b}	69.23
Group B vitamins (mg/100 g DW)	Pyridoxine	3.14±0.18^{a}	1.01±0.55^{b}	67.83	0.85±0.03^{c}	72.92	0.75±0.18^{c}	76.11
	Niacin	0.12±0.02^{a}	0.09±0.06^{b}	25	0.05±0.01^{b}	58.33	Nd	100
	Thiamine	0.30±0.04^{a}	0.18±0.01^{b}	40	0.16±0.01^{b}	46.66	0.14±0.02^{c}	53.33
Organic acids (g/100 g DW)	Tartaric acid	11.06±0.23^{a}	10.67±0.43^{ab}	3.52	10.54±0.27^{ab}	4.70	10.35±0.14^{b}	6.41
	Citric Acid	6.74±0.37^{a}	6.58±0.21^{ab}	2.37	6.39±0.11^{ab}	5.19	6.02±0.08^{b}	10.68
	Malic Acid	8.62±0.05^{a}	8.59±0.17^{ab}	0.34	8.42±0.08^{ab}	2.32	8.11±0.04^{b}	5.91
Phenolic compounds (g/100 g DW)	Chlorogenic acid	Nd	Nd	Nd	Nd	Nd	Nd	Nd
	Ellagic acid	0.64±0.03^{a}	0.47±0.01^{b}	26.56	0.43±0.12^{b}	32.81	0.25±0.07^{c}	60.93
	p-Coumaric acid	0.57±0.17^{a}	0.21±0.03^{b}	63.15	0.33±0.07^{b}	42.10	0.13±0.04^{c}	77.19
	Caffeic acid	0.64±0.05^{a}	0.59±0.01^{a}	7.81	0.47±0.03^{bc}	26.56	0.41±0.06^{c}	35.93
	Rutin	0.26±0.02^{a}	0.11±0.04^{b}	57.69	0.03±0.01^{c}	88.46	0.09±0.01^{b}	65.38
Resveratrol (g/100 g DW)		1.26±0.05^{a}	0.31±0.03^{b}	75.02	0.30±0.01^{b}	75.98	0.21±0.01^{c}	82.90

*Nd: Not detectable. TPC: Total phenolic content. AC: Antioxidant capacity. Different letters on the same line indicate statistical difference ($p < 0.05$).

Determination of kinetic parameters

Kinetic parameters of trans-resveratrol

Thermal degradation of *trans*-resveratrol in gilaburu fruit was examined at (50, 60, and 70) °C. Arrhenius plots and the first-order reaction model obtained for *trans*-resveratrol during hot-air drying of gilaburu fruits at different temperatures are shown in Figure 3. As seen in Figure 3, the thermal degradation of *trans*-resveratrol in dried gilaburu fruits fitted the first-order kinetic model.

The values of k, $t_{1/2}$, Q_{10}, and E_a of the *trans*-resveratrol in dried gilaburu fruits are shown in Table 7.

The activation energy (E_a) indicates the reaction's sensitivity to temperature, which refers to the energy required to activate the reaction. Likewise, the reaction rate constant (k) value also indicates the thermal sensitivity of the reaction. The Q_{10} value represents the effect of every 10 °C change on the reaction, and the $t_{1/2}$ value represents the half-life of the reaction. The k value was increased with increasing drying temperature. The highest k value was identified at 70 °C. A decrease in the $t_{1/2}$ value was observed due to increased temperature. The lowest $t_{1/2}$ was 2.58 hours at 70 °C. The highest Q_{10} value was 4.25 for the studied temperature range (60 °C –70 °C).

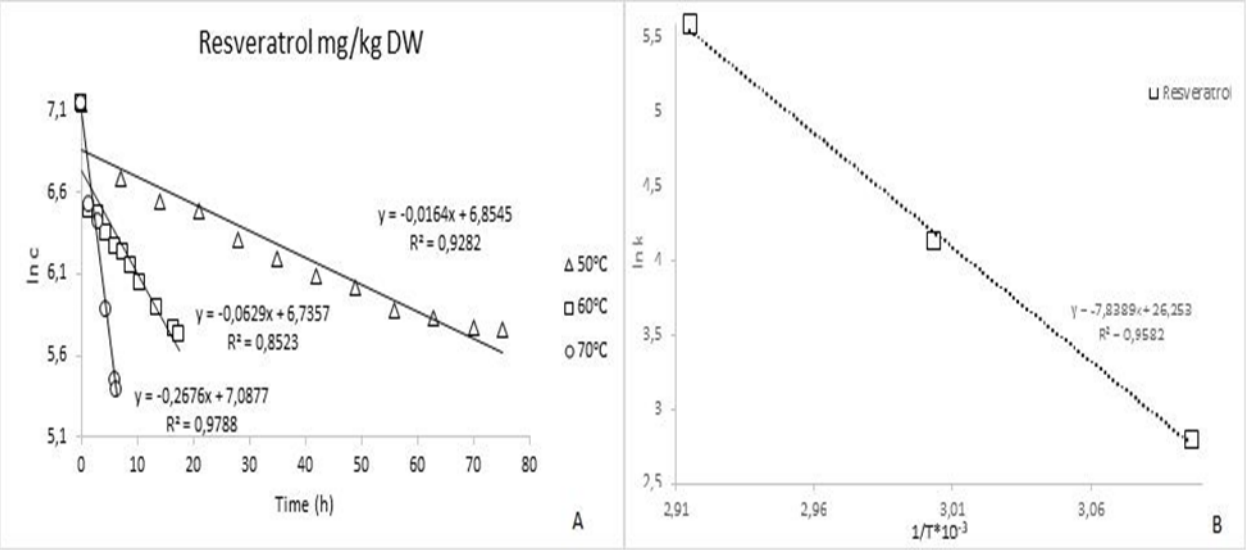

Figure 3. First order plots (A) and Arrhenius plots (B) of trans-resveratrol during drying of gilaburu fruits.

Kinetic parameters of water-soluble vitamins

Thermal degradation of water-soluble vitamins of gilaburu fruits was analyzed at (50, 60, and 70) °C. Thermal degradation of water-soluble vitamins fitted the first-order kinetic model (Figure 4). Thermal degradation of ascorbic acid and thiamine fit the first-order kinetic model in different dried fruits during hot-air drying [75].

The Arrhenius plots obtained for the thermal degradation of water-soluble vitamins during the drying of gilaburu fruits in different temperatures are presented in Figure 5.

The kinetic parameter values of water-soluble vitamins are shown in Table 6. The k value of all vitamins increased with increasing temperature. The lowest rate constant value was calculated for ascorbic acid. The $t_{1/2}$ value decreased depending on the increase in temperature. This result indicates that vitamins decompose more at high temperatures. The lowest $t_{1/2}$ value for water-soluble vitamins was at 70 °C.

For all water-soluble vitamins in gilaburu fruits, the Q_{10} value was calculated for temperatures between 50 °C and 70 °C. The highest Q_{10} value for ascorbic acid, thiamine, and pyridoxine was calculated at (50–60) °C while for niacin at (60–70) °C. This result shows that the decomposition reaction gets more affected by the changes in temperature.

As shown in Table 6, niacin has the highest E_a value (40.45 kcal mol^{-1}); the rest were thiamine, ascorbic acid, and pyridoxine.

Kinetic parameters of TPA and AC

The present study first reported data on the thermal degradation of TPC and AC of gilaburu fruits. Thermal degradation of TPC and AC followed the first-order kinetic model. Similarly, Tepe and Ekinci [41] have reported a first-order reaction of jujube fruits during hot drying. First-order graphics of TPC (A) and AC (B) are presented in Figure 6, while Arrhenius graphs of TPC and AC during hot air drying of gilaburu fruits at different temperatures are shown in Figure 7.

The degradation kinetics data of TPC and AC are given in Table 7. The k value of TPC and AC increased

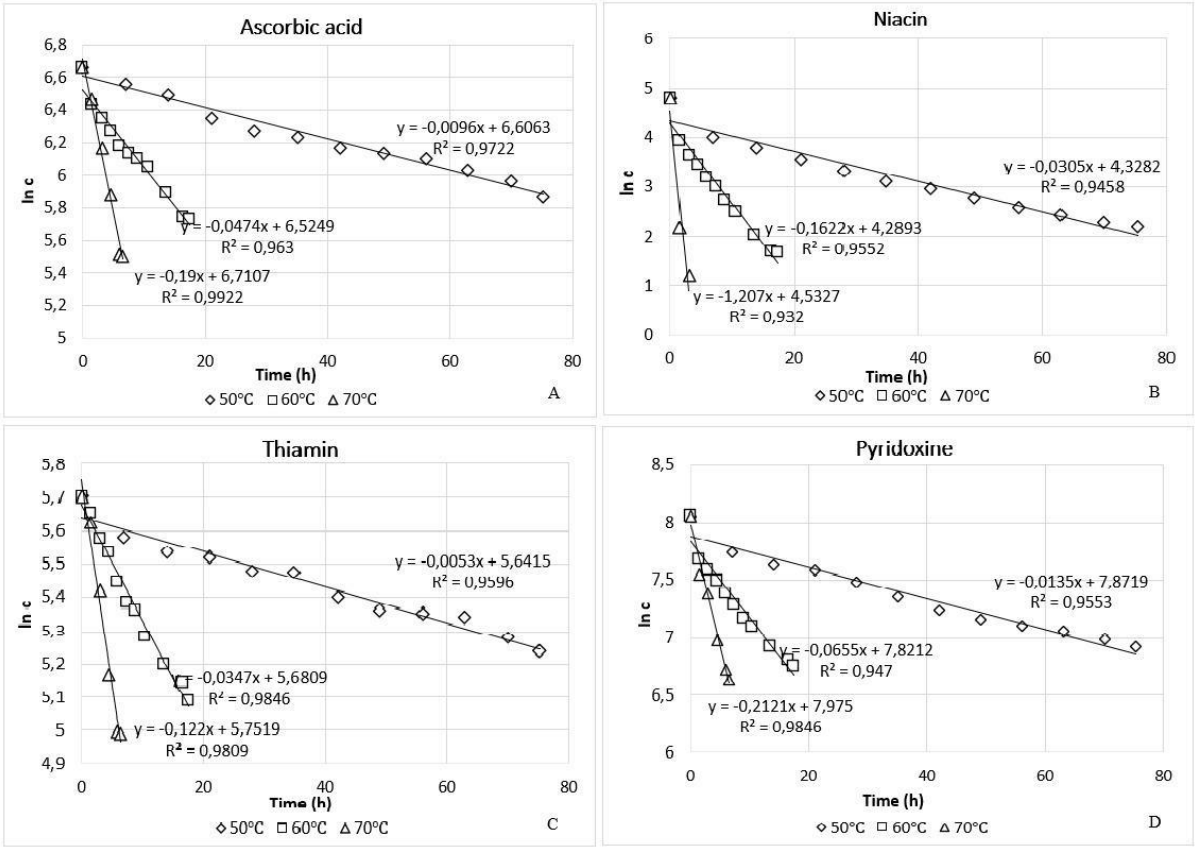

Figure 4. First-order kinetics of ascorbic acid (A), niacin (B), thiamine (C), and pyridoxine (D) of dried gilaburu fruits.

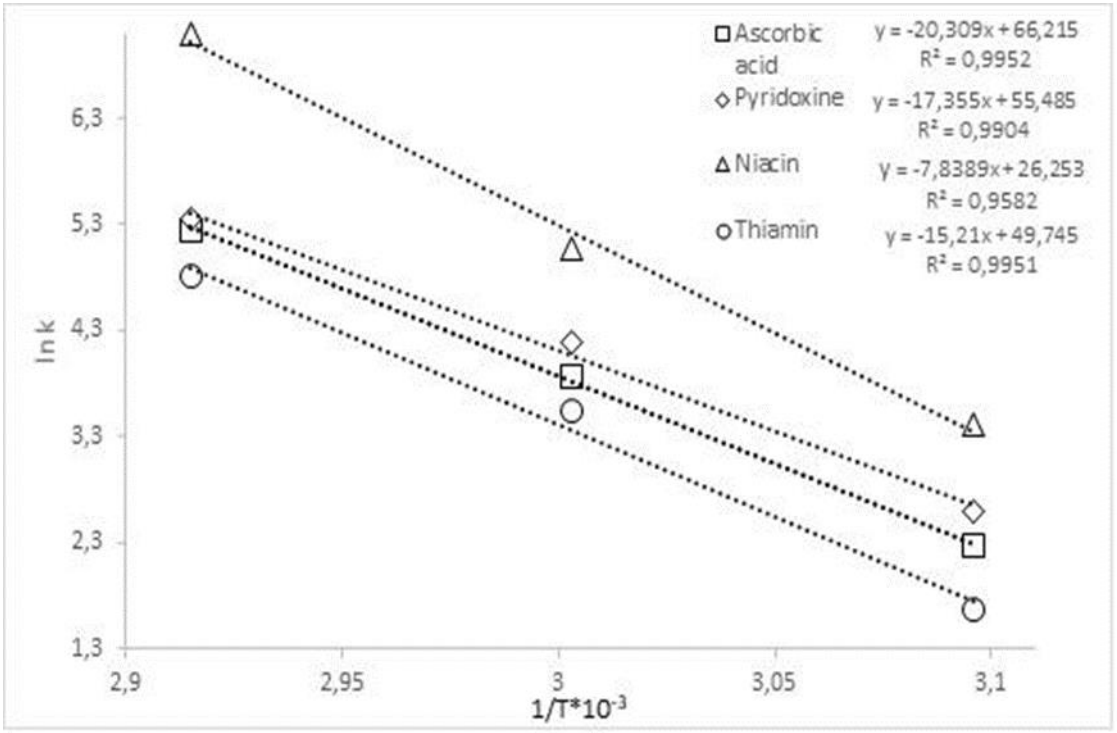

Figure 5. Arrhenius plots of water-soluble vitamins of dried gilaburu fruits.

Table 6. First-order kinetic parameters of water-soluble vitamins.

	T (°C)	k (h^{-1})	$t_{1/2}$ (h)	D (h)	Q_{10} (50–60) °C	Q_{10} (60–70) °C	E_a (kcal mol^{-1})	E_a (kJ mol^{-1})
	50	0.0096	72.18	239.89				
Ascorbic acid	60	0.0474	14.62	48.58	4.93	4.00	32.87	137.53
	70	0.1900	3.64	12.12				
	50	0.0305	22.72	75.50				
Niacin	60	0.1622	4.27	14.19	5.31	7.44	40.45	169.27
	70	1.207	0.57	1.90				
	50	0.0053	130.75	434.52				
Thiamine	60	0.0347	19.97	66.36	6.54	3.51	34.57	144.68
	70	0.1220	5.68	18.87				
	50	0.0135	51.33	170.59				
Pyridoxine	60	0.0655	10.58	35.16	4.85	3.23	30.30	126.80
	70	0.02121	3.26	180.85				

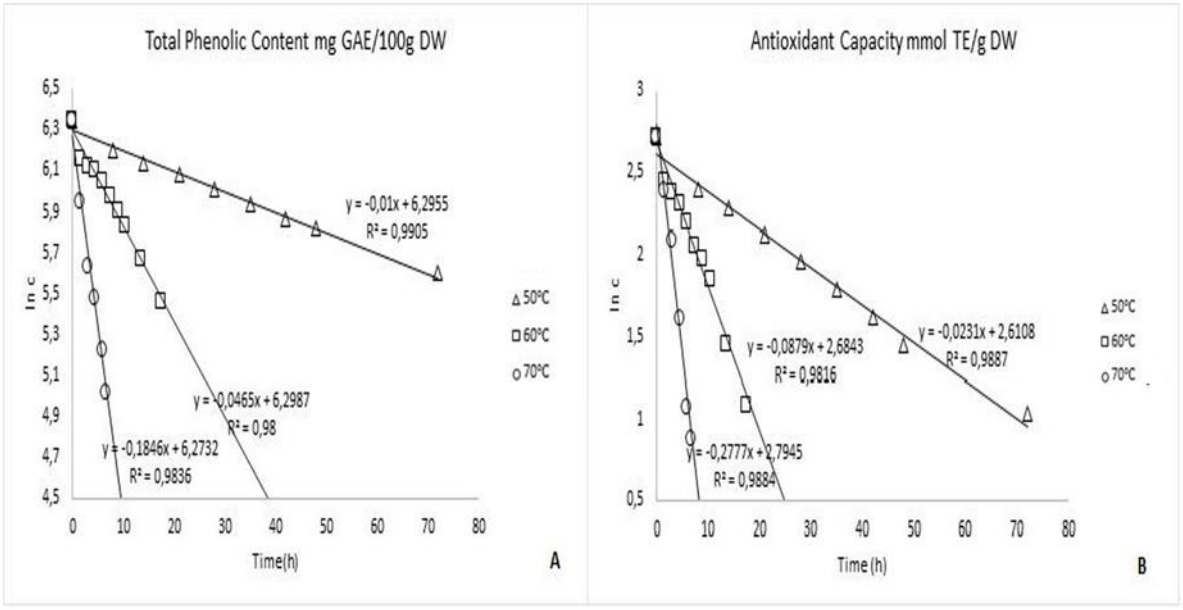

Figure 6. First-order kinetics of TPC (A) and AC (B) of dried gilaburu fruits.

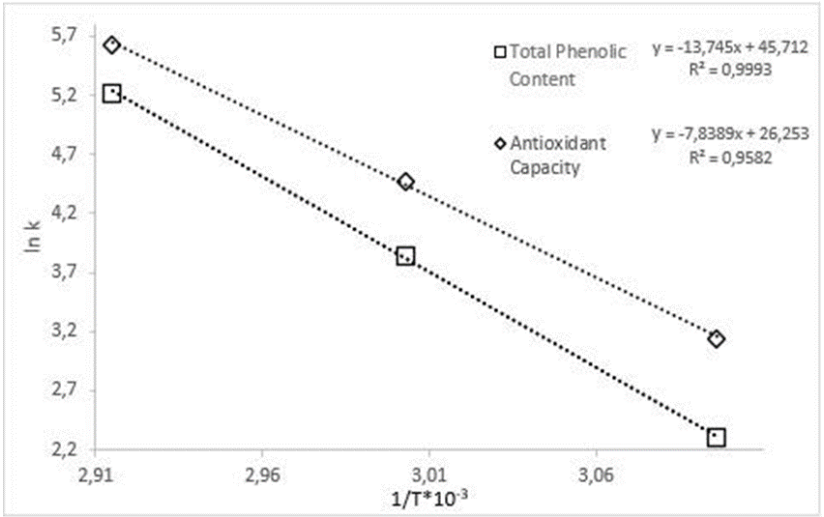

Figure 7. Arrhenius plots of TPC and AC of dried gilaburu fruits.

depending on the increment in temperature. Accordingly, a decrease in the $t_{1/2}$ value was observed. The highest D value was calculated at 50 °C. The highest Q_{10} value of TPC and AC content was 4.65 and 3.80 hours, respectively, at 50-60 °C. E_a values were 32.10 kcal mol^{-1} for TPC and 27.38 kcal mol^{-1} for AC.

The Q_{10} values from 50°C to 60 °C and from 60 °C to 70 °C were found to be 1.53 and 2.17, respectively. On the other hand, the high Q_{10} value between (60–70) °C shows that the thermal degradation of AC is more sensitive in this range than the increase between (50–60) °C.

Table 7. First-order kinetic parameters of TPC, AC and resveratrol.

	T (°C)	k (h^{-1})	$t_{1/2}$ (h)	D (h)	Q_{10} (50–60) °C	Q_{10} (60–70) °C	E_a (kcal mol^{-1})	E_a (kJ mol^{-1})
TPC	50	0.010	69.3	230.3	4.65	3.96	32.10	134.31
	60	0.0465	14.90	14.19				
	70	0.1846	3.75	1.90				
AC	50	0.0231	30.00	99.69	3.80	3.15	27.38	114.57
	60	0.0879	7.88	26.20				
	70	0.2777	2.49	8.29				
Trans-resveratrol	50	0.0164	42.25	140.42	3.83	4.25	30.71	128.50
	60	0.0629	11.01	36.61				
	70	0.2676	2.58	8.60				

CONCLUSION

The study shows that the drying temperature significantly affects the drying and moisture ratio of gilaburu fruits due to the drying process losses in TPC, AC, organic acids, water-soluble vitamins, phenolic components, and *trans*-resveratrol content. While the highest loss rates were observed at 70 °C, it was revealed that the components in the gilaburu fruits were better preserved as a result of the drying process at 50 °C. Therefore, when evaluated in terms of quality losses, it was observed that the best drying temperature was 50 °C. The degradation reaction at all compounds was carried out per the first-order kinetic model. In addition, further research should be conducted on different drying methods and pre-treatment (such as immersing citric acid and ethanol solution, hot water blanching, and ultrasound) in addition to hot air drying to ensure less loss of components of gilaburu fruits and a shorter drying time. Moreover, color kinetics can be inspected with dried fruits. Consequently, the drying data of gilaburu fruit obtained by this study has created an alternative to the different evaluation of gilaburu fruit consumed only as brine and fruit juice.

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KINETIKA SUŠENJA I RAZGRADNJE BIOAKTIVNIH NUTRITIVNIH JEDINJENJA PLODA CRVENE KALINE (*Viburnum opulus* L.)

Ovaj rad ima za cilj da utvrdi da li je sušenje pogodna metoda konzervisanja ploda crvene kaline (Viburnum opulus L.) i kakve su promene u njegovim bioaktivnim komponentama na kraju procesa sušenja. U ovoj studiji, plodovi crvene kaline su sušeni u sušari na različitim temperaturama (50, 60 i 70) °C. Analize trans-resveratrola, vitamina rastvornih u vodi, organskih kiselina i fenolnih jedinjenja vršene su HPLC metodom, a sadržaj ukupnih fenola i antioksidaciona aktivnost spektrofotometrijski. Kao rezultat sušenja plodova crvene kaline na (50, 60 i 70) °C, najveći gubitak komponenti zabeležen je na 70 °C, pri čemu gubici u sadržaju ukupnih fenola i antioksidacione aktivnosti ploda crvene kaline nakon sušenja na 70 °C iznose 73,64% i 84,08%, redom. Sadržaj trans-resveratrola je bio 1,26±0,05 (g/100 g suve mase u svežem voću), a smanjio se na 0,31±0,03, 0,30±0,01 i 0,21±0,01 nakon sušenja na (50, 60 i 70) °C, redom. Što se tiče vitamina, najveći gubitak je zabeležen u niacinu. Sadržaj askorbinske kiseline, piridoksina, niacina i tiamina u svežem plodu crvene kaline smanjen je nakon sušenja na 50°C, 60 °C i 70 °C. Pored toga, modelovana je kinetika sušenja u pogledu vitamina rastvornih u vodi, sadržaja ukupnih fenola, antioksidacione aktivnosti i trans-resveratrol. Model Pejdža najbolje opisuje kinetiku sušenja voća na 70 ℃, a parabolnki model na 50 ℃ i 60 ℃. Termička degradacija vitamina rastvornih u vodi, sadržaja ukupnih fenola, antioksidacione aktivnosti i trans-resveratrola prati kinetički model prvog reda.

Ključne reči: antioksidacioni kapacitet, kinetika sušenja, gilaburu, trans-resveratrol, sadržaj ukupnih fenolna, vitamini rastvorni u vodi.

NAUČNI RAD