SCIENTIFIC PAPER

Hot-Air Drying and Degradation Kinetics of Bioactive Compounds of Gilaburu (*Viburnum opulus* L.) Fruit

Aslı DÖNMEZ, Çetin KADAKAL*

Department of Food Engineering, Faculty of Engineering, Pamukkale University, Denizli, Turkey

Received 14.6.2022

Revised 27.3.2023

Accepted 8.6.2023

*Corresponding Author, E-mail: ckadakal@pau.edu.tr
Address: Department of Food Engineering, Faculty of Engineering, Pamukkale University, Denizli, Turkey Tel: +90 5301104629

Abstract

The purpose of this study is to determine whether drying is a suitable preservation method for gilaburu fruit and to determine 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 cabinet dryer at different temperatures (50,60 and 70 °C). The analyses of *trans*-resveratrol, water-soluble vitamins, organic acids and phenolic compounds were made by using the HPLC method while total phenolic contents and antioxidant activity were spectrophotometric. As a result of drying of gilaburu fruit at 50, 60 and 70 °C, the highest component loss was observed at 70 °C. A loss of 73.64% and 84.08%, respectively, was 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, 60 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 from 0.78, 3.14, 0.12 and 0.30 g/100 g to 0.24, 0.75, 0 and 0.14 g/100 g, respectively, after drying at 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 the first-order kinetic model.

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

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 best predict the experimental drying.

INTRODUCTION

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 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 have 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 cranberrybush, cranberry tree in the world, 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 ripe fruits of the gilaburu plant are bright red in color, round-oval in shape, single-seeded, thin-shelled and juicy. Ripe fruits are acidic and have bitter taste [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 in the form of 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, diuretic, 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 and also contains vitamins A, E, micronutrients (Cu, Mn, Fe and Zn) and macro nutrients (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, which is a phenolic acid in *trans*-isomer structure, has many health benefits since it is such as 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 the sun drying, there is minimal loss of nutritional value and better physical preservation thanks to adjustable time and temperature parameters, 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, there are studies investigating 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 as a result of 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 the use of industrial purposes. In addition, determining the drying characteristics and creating mathematical models to determine the most suitable drying parameters at different temperatures are targeted.

MATERIALS 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 fruits to be used in the analysis were selected from ripe ones. 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. Tray cabinet dryer consisted of a resistance heater providing the temperature, a temperature control panel and a fan providing the air flow (EUC442 model, ENDA, Turkey). The cabin, which has dimensions of 70 cm x 55 cm x 100 cm, operates in the temperature range of 40-120 °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 in total,

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, whose temperature and time parameters determined, were distributed homogeneously on the drying trays (25 cm x 20 cm x 3 cm) as 2000 g. Average air velocity and relative humidity of 2 m/s and 20% were recorded, respectively. Gilaburu fruits were spread homogeneously as single layer on the drying tray. During the drying process, fruits were weighed every half hour for the first 5 hours and then at 1-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 the calculation of mathematical models. The equation, used for the calculation of the humidity ratio, is given using Equation (1).

$$\mathrm{MR} = \frac{\mathrm{M}_t - M_e}{M_i - M_e} \quad (1)$$

MR : moisture ratio of samples (dimensional)

M_i : Initial moisture content of sample (g water g^{-1} DW)

M_t : moisture content of sample for any time (g water g^{-1} DW)

M_e : Equilibrium moisture content of example at t time (g water g^{-1} DW)

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

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

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

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

M_t: moisture content of sample for any time (g water g^{-1} DW)

$M_{t+\Delta t}$:moisture content of sample at any t+Δt time (g water g^{-1} DW)

Δt: Time difference between two measurements (hours)

Mathematical models are used to 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. In order to determine the best model to be used in explaining the relationship between experimental data and predicted data, the model with the highest R^2 value and the lowest $\chi 2$ and RMSE should be selected. MATLAB (R2015a) program was used to calculate the mathematical modeling data. The mathematical models used in this study are given in Table 1.

Table 1.

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

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

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

$MR_{pre,i}$: Predicted moisture ratio

$MR_{exp,i}$: Experimental moisture ratio

N: Number of experimental data

n: Constants of thin layer drying models

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 Equation 6 to calculate the effective moisture diffusion in spherical products, provided that there is no shrinkage in the dried material and the effective diffusion is constant [41].

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

D_{eff}: Effective moisture diffusivity ($m^2\ s^{-1}$)

r: Arithmetical average of radius of samples at measured intervals (m).

Equation 6 was shortened to form Equation 7 [37].

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

The slope of the graph drawn by Equation 7 was calculated by Equation 8.

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

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

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

R: Universal gas constant (8.314 J mol^{-1} K^{-1} or 1.987 cal mol^{-1} K^{-1}

T: Actual temperature (K)

E_a: Activation energy (kJ mol^{-1} or kcal mol^{-1})

D_0: Constant before exponential ($m^2\ s^{-1}$)

Equation 10 is obtained by using the natural logarithm of Equation 8 and Equation 9.

$$lnD_{eff} = lnD_0 - \frac{E_a}{RT} \quad (10)$$

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

Analysis of *trans*-resveratrol, a phenolic component, was carried out according to the method suggested by Singh and Pai [43]. Methanol was used for the extraction of gilaburu fruits which are dried at different temperature parameters. HPLC device (SHIMADZU LC20AD), consist of column oven (SHIMADZU CTO-20A), column (ACE C18 (7.8x300 mm)), pump

(SHIMADZU LC-20AD), degasser (SHIMADZU DGU-20A3) and photo diode 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

HPLC device (SHIMADZU LC20AD) was used for the analysis of 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 with the equation obtained from the calibration curve with 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 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 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 used for the final extraction of 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 is 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 equation of the calibration curve 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.

Table 2.

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 to be used in the calculations was prepared by using the standards of 25, 50, 75 and 100 mg/L. 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 for the determination of 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 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 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 concentrations.

Statistical analysis

SPSS software statistical package program (SPSS ver. 23, SPSS Inc., Chicago, IL, USA) was used for statistical analysis of the data. One way analysis of variance (ANOVA) was used to evaluate differences between treatments with the significance level $p<0.05$. Duncan 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

Moisture ratio (A) and drying rate (B) of gilaburu fruits during hot air drying are shown in Figure 1. At the beginning, 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 during the drying of the gilaburu fruits with hot air. Accordingly, the drying time was reduced and found to be 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 Kayseri region, the samples were dried at an air velocity of 1.3 m/s. The drying process at 60, 70 and 80 °C 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 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].

Fig. 1

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

Table 3.

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

In Table 4, D_{eff} and E_a values of gilaburu fruits are presented. 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 that the drying process is fast [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.

Table 4.

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, E_a value determined 133.81kJ mol^{-1}. In a study conducted with goji berry fruit, which is similar to gilaburu fruit, the E_a value was found to be 48.37 kJ mol^{-1} [42]. It is thought that the differences between E_a values might be due to different drying conditions and fruit types. D_{eff} values obtained as a result of drying gilaburu fruit increased depending on the temperature increase. In a study with grape, the drying process of grapes at 30, 35, 40 and 45 °C was examined and it was stated that the highest D_{eff} value was at 45 °C, which is the highest drying temperature [54]. It was seen that the analyzed data supported our study. Arrhenius relation between D_{eff} and T^{-1} presented in Figure 2.

Fig. 2

The effect of the drying process

Changes in *trans*-resveratrol contents

In this study, *trans*-resveratrol content of fresh fruits was determined as 1.26 g/100 g. A decrease in *trans*-resveratrol content was observed due to 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, spray drying process was applied and the effect of 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 obtained were compared, it was observed that the temperature application caused a decrease in *trans*-resveratrol. The reason for this decrease is due to the fact that *trans*-resveratol is a lipophilic polyphenol sensitive to thermal degradation [56].

Changes in water-soluble vitamin contents

Preservation of heat sensitive vitamins during the drying process is considered as an indicator of food quality [57]. Especially, the ascorbic acid is a critical quality parameter. In general, if the fact that 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 pyridoxine. Pyridoxine was also dominant vitamin in doum fruit [59]. A significant decrease in vitamin content was observed in parallel with the increase in drying temperature.

Ascorbic acid content of fresh gilaburu fruit was determined as 0.78 g/100 g DW. In a study done by Akbulut et al., it was stated that 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]. The reason for this decrease in the ascorbic acid content of fruits is due to the low thermal sensitivity of ascorbic acid [56]. In the current study, pyridoxine content of fresh gilaburu fruits was determined as 3.14 g/100 g DW. After the drying process at 70°C, pyridoxine content was decreased and determined as 0.75 g/100 g DW. The thiamine content decreased from 0.30 g/100 g DW to 0.14 g/100 g DW after drying at 70°C. Heat treatment applied at high temperature 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 g/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 on the investigation of the vitamin B content of gilaburu fruit. In a study conducted with different date varieties, it was reported that the date fruit contains thiamine, riboflovin, niacin, pantetonic acid and pyridoxine [59]. In a similar study with caper fruit, these types of vitamins were found [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 riboflovin after drying the rosehip fruit [64]. In a similar study, it was determined that there was a decrease in vitamin values as a result of drying the jujube fruit at different temperatures [65].

Changes in organic acids contents

It was determined that the dominant organic acid of gilaburu fruit was tartaric acid. In addition, it was determined that the fresh fruit contains malic and citric acids. In a study examining the organic acid content of gilaburu fruit, it was reported that the dominant organic acid was tartaric acid [1]. Tartaric, citric and malic acid contents of fresh gilaburu fruit were detected to be 11.06±0.23, 6.74±0.37 and 8.62±0.05, respectively. In a study conducted with 11 different gilaburu samples, which are grown in different parts of the country, the amount of malic acid was found to be between 578.0 and 2090.0 mg/100 g. In the same study, it was

reported that the amount of citric acid was in the range of 270.0 - 1630.0 mg/100 g [66]. In another study, it was reported that 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. The reason for this situation is thought to be factors such as species diversity, climatic conditions and soil properties that affect the composition.

The changes in organic acid content after drying of 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 determined as 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 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 of drying temperature. Adiletta et al. stated that the organic acid amount of red and white grapes decreased after drying at 50°C [68]. The reason for the decrease in organic acids can be explained by temperature 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 found to be caffeic acid and ellagic acid. After drying at all temperatures, the amount of phenolic compounds decreased. While the value of caffeic acid, which is the dominant phenolic component, was 0.64 g/100 g DW in fresh fruits, that value 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, it was reported that the chlorogenic, caffeic and *p*-coumaric acid contents were in the range 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 part of the gilaburu fruit 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, it was observed that long-term heat treatment applied at high temperatures destroyed the

phenolic compounds [71]. It is thought that this is due to the fact that phenolic compounds have an easily oxidized structure and heat treatment application causes a decrease in the content of phenolic compounds [72].

Changes in total phenolic content and antioxidant capacity

Total phenolic content and antioxidant capacity values of gilaburu fruit is shown in Table 5. The total phenolic content of gilaburu fruits was decreased by the increase in drying temperature. 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 reason for the decrease in antioxidant activity as a result of the drying process is the losses in antioxidant compounds during drying [73]. Zarifikhosroshahi [17] determined the TPC value as 1009.89 mg GAE/100 g DW in gilaburu samples belonging to the Kayseri region. This value was found to be higher than the value obtained as a result of our analysis. In another study, conducted with gilaburu fruits obtained from Kayseri region, TPC value was determined as 633.56 mg GAE/100 g DW [74] and this value is close to the result obtained in our analysis. The antioxidant capacity of fresh fruit was detected 15.08 µmol TE/g DW and it decreased to 2.40 µmol TE/g DW after drying process at 70 °C.

Table 5.

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, thermal degradation of *trans*-resveratrol in dried gilaburu fruits fitted the firs-order kinetic model.

Fig. 3

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 and this value refers to the energy required to activate the reaction. Likewise, 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 $t_{1/2}$ value was observed due to an increase in temperature. The lowest $t_{1/2}$ was determined as 2.58 hours at 70 °C. The highest Q_{10} value was found as 4.25 for the studied temperature range (60-70°C).

Kinetic parameters of water-soluble vitamins

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

Fig. 4

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

Fig. 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 in ascorbic acid. The $t_{1/2}$ value decreased depending on the increase in temperature. This result indicates that vitamins decompose more at high temperatures. It was determined that the lowest $t_{1/2}$ value for water-soluble vitamins was at 70 °C.

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

When the E_a values in Table 6 were examined the highest value belonged to niacin (40.45 kcal mol-1) and the rest were thiamine, ascorbic acid and pyridoxine in order.

Table 6.

Kinetic parameters of TPA and AC

This is the first study reporting data on the thermal degradation of TPC and AC of gilaburu fruits. It was found that 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 the hot drying process. First-order graphics of TPC (A) and AC (B) are presented

in Figure 6 and Arrhenius graphs of TPC and AC during hot air drying of gilaburu fruits at different temperatures are shown in Figure 7.

Fig. 6

Fig. 7

The degradation kinetics data of TPC and AC are given in Table 7. The k value of TPC and AC increased depending on the increment in temperature. Accordingly, a decrease in $t_{1/2}$ value was observed. The highest D value for both temperatures was calculated at 50°C. The highest Q_{10} value of TPC and AC content was found to be 4.65 and 3.80 hour, respectively, at 50-60 °C. E_a values were found as 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.

CONCLUSIONS

The study shows that the drying temperature has a significant effect on the drying and moisture ratio of gilaburu fruits. As a result of the drying process losses occurred 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 were carried out in accordance with 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, ultrasound) in addition to hot air drying in order 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, which is consumed only as brine and fruit juice.

CONFLICT OF INTEREST

There are no conflicts to declare.

AUTHOR CONTRIBUTIONS

Aslı DÖNMEZ: investigation, writing-original draft, review and editing.

Çetin KADAKAL: conceptualization, methodology, visualization, writing-original draft, review and editing.

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Table Captions

Table 1. Mathematical models

Table 2: Methods used in chromatographic analyses

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

Table 4. Effective moisture diffusivity and activation energy of gilaburu

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

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

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

Figure Legends

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

Fig. 2. Arrhenius type relation between D_{eff} and T^{-1}

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

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

Fig. 5. Arrhenius plots of water soluble vitamins of dried gilaburu fruits

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

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

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]

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	ACE C18	0.8 ml/min	40°C	324 nm	0.1 M KH_2P0_{4+}0.1 M KOH
	Niacin	ACE C18	0.8 ml/min	40°C	261 nm	0.1 M KH_2P0_{4+}0.1 M KOH
	Thiamin	ACE C18	0.8 ml/min	40°C	234 nm	0.1 M KH_2P0_{4+}0.1 M KOH
Organic Acids	Tartaric Acid	ACE C18	1.0 ml/min	25°C	214 nm	0.01 N H_2SO_4
	Citric Acid	ACE C18	1.0 ml/min	25°C	214 nm	0.01 N H_2SO_4
	Malic Acid	ACE C18	1.0 ml/min	25°C	214 nm	0.01 N H_2SO_4
Phenolic Compounds	Chlorogenic acid	ACE C18	0.5 ml/min	25°C	280 nm	0.1 orto-H_3PO_4:C_2H_3N
	Ellagic acid	ACE C18	0.5 ml/min	25°C	254 nm	0.1 orto-H_3PO_4:C_2H_3N
	p-Coumaric acid	ACE C18	0.5 ml/min	25°C	280 nm	0.1 orto-H_3PO_4:C_2H_3N
	Caffeic acid	ACE C18	0.5 ml/min	25°C	280 nm	0.1 orto-H_3PO_4:C_2H_3N
	Rutin	ACE C18	0.5 ml/min	25°C	360 nm	0.1 orto-H_3PO_4:C_2H_3N
Trans-resveratrol		ACE C18	0.8 ml/min	30°C	306 nm	Metanol:10mM KH_2PO_4: C_2H_3N

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	50°C	a= -0.0003746	b= 0.00000003662		0.00015759	0.0124	0.9985
Singh	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 fruit

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

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
Water-soluble vitamins (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
	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	0	100
	Thiamin	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

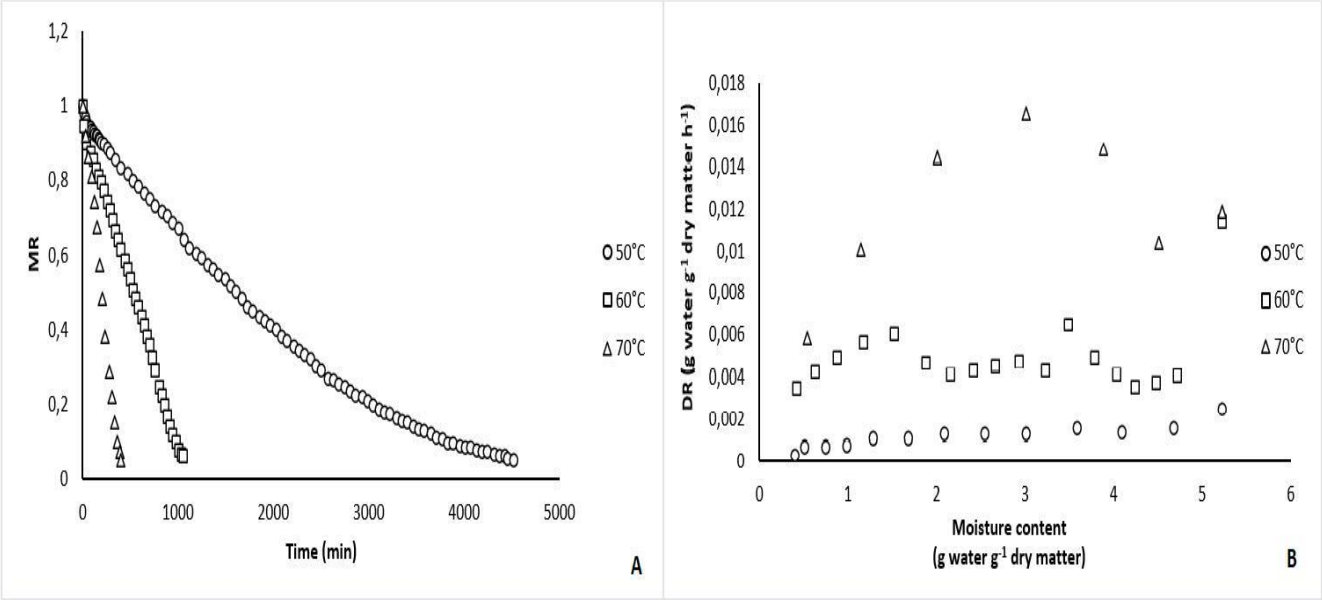
*Nd: Not detectable. TPC: Total phenolic content. AC: Antioxidant capacity. Different letters on the same line indicate statistical difference ($p < 0.05$).

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})
Ascorbic acid	50	0.0096	72.18	239.89	4.93	4.00	32.87	137.53
	60	0.0474	14.62	48.58				
	70	0.1900	3.64	12.12				
Niacin	50	0.0305	22.72	75.50	5.31	7.44	40.45	169.27
	60	0.1622	4.27	14.19				
	70	1.207	0.57	1.90				
Thiamine	50	0.0053	130.75	434.52	6.54	3.51	34.57	144.68
	60	0.0347	19.97	66.36				
	70	0.1220	5.68	18.87				
Pyridoxine	50	0.0135	51.33	170.59	4.85	3.23	30.30	126.80
	60	0.0655	10.58	35.16				
	70	0.02121	3.26	180.85				

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				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0,6
0,4
0,2
0
MR
0
1000
2000
3000
4000
5000
Time (min)
50°C
60°C
70°C
A
0,018
0,016
0,014
0,012
0,01
0,008
0,006
0,004
0,002
0
DR (g water g-1 dry matter h-1)
0
1
2
3
4
5
6
Moisture content
(g water g-1 dry matter)
50°C
60°C
70°C
B

Figure 1

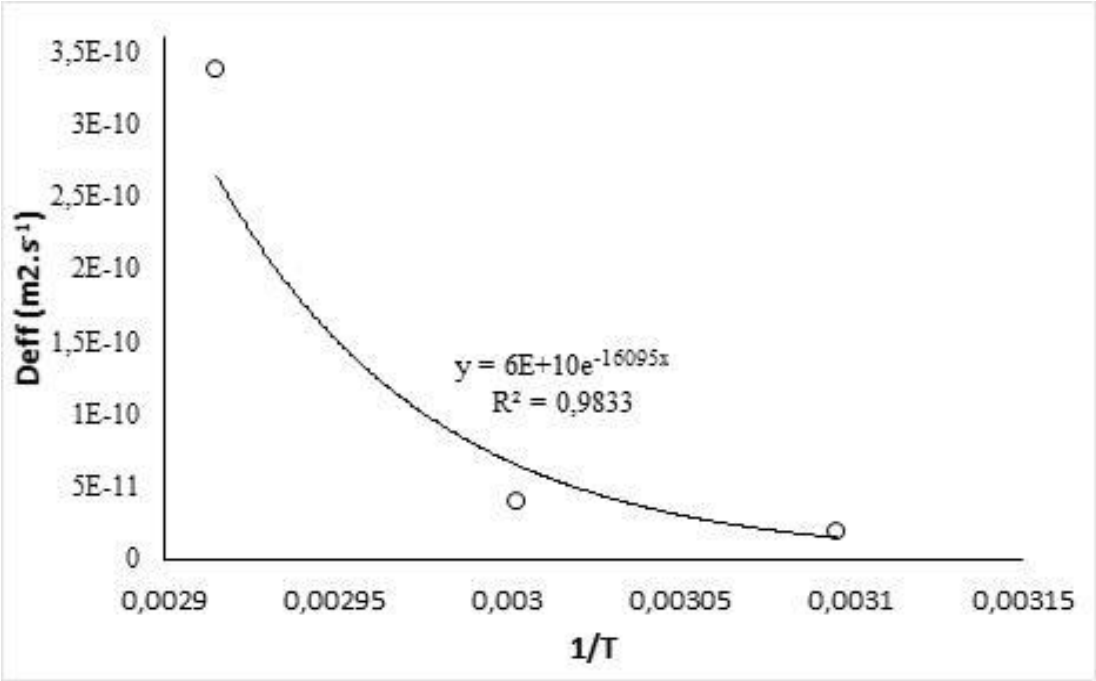
3,5E-10
3E-10
2,5E-10
2E-10
1,5E-10
1E-10
5E-11
0
Deff (m2.s-1)
y = 6E+10e-16095x
R² = 0,9833
0,0029
0,00295
0,003
0,00305
0,0031
0,00315
1/T

Figure 2

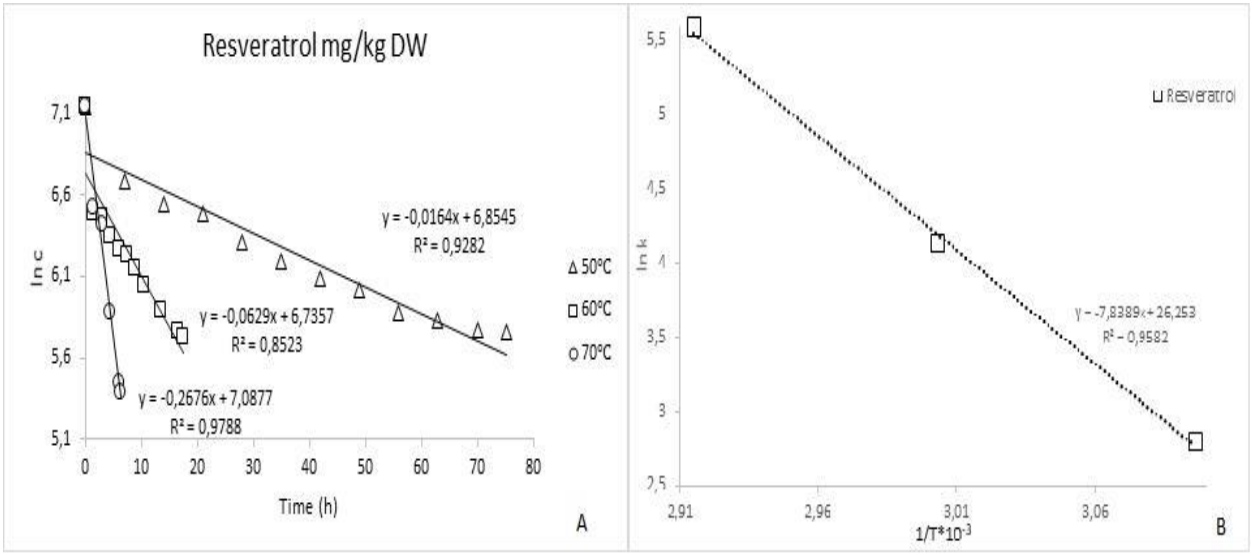
Resveratrol mg/kg DW
ln c
Time (h)
y = -0,0164x + 6,8545
R² = 0,9282
y = -0,0629x + 6,7357
R² = 0,8523
y = -0,2676x + 7,0877
R² = 0,9788
50°C
60°C
70°C
A
Resveratrol
ln k
1/T*10-3
y = -7,8389x + 26,253
R² = 0,9582
B

Figure 3

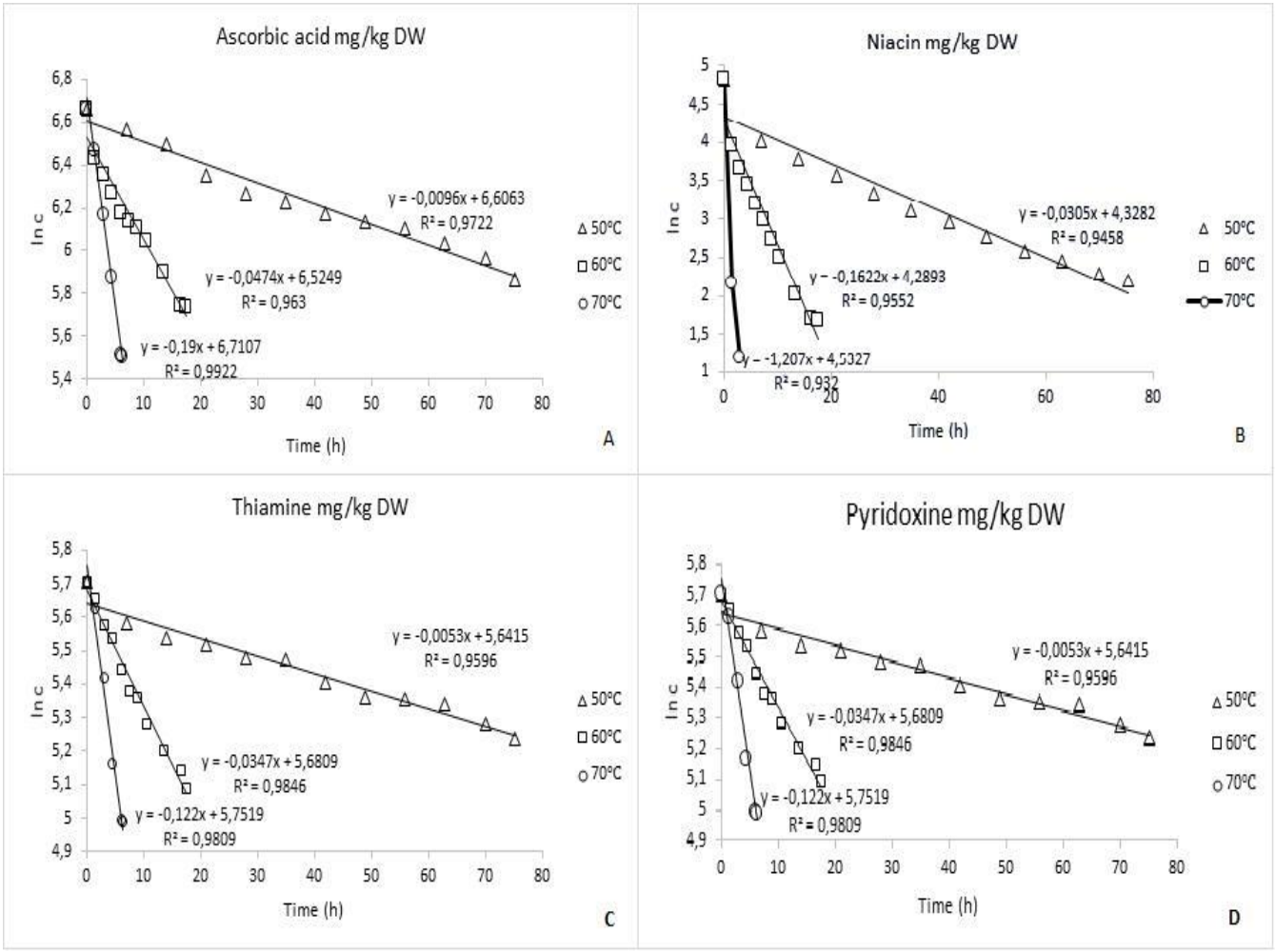
Ascorbic acid mg/kg DW
ln c
Time (h)
y = -0,0096x + 6,6063
R² = 0,9722
y = -0,0474x + 6,5249
R² = 0,963
y = -0,19x + 6,7107
R² = 0,9922
50°C
60°C
70°C
A
Niacin mg/kg DW
ln c
Time (h)
y = -0,0305x + 4,3282
R² = 0,9458
y = -0,1622x + 4,2893
R² = 0,9552
y = -1,207x + 4,5327
R² = 0,932
50°C
60°C
70°C
B
Thiamine mg/kg DW
ln c
Time (h)
y = -0,0053x + 5,6415
R² = 0,9596
y = -0,0347x + 5,6809
R² = 0,9846
y = -0,122x + 5,7519
R² = 0,9809
50°C
60°C
70°C
C
Pyridoxine mg/kg DW
ln c
Time (h)
y = -0,0053x + 5,6415
R² = 0,9596
y = -0,0347x + 5,6809
R² = 0,9846
y = -0,122x + 5,7519
R² = 0,9809
50°C
60°C
70°C
D

Figure 4

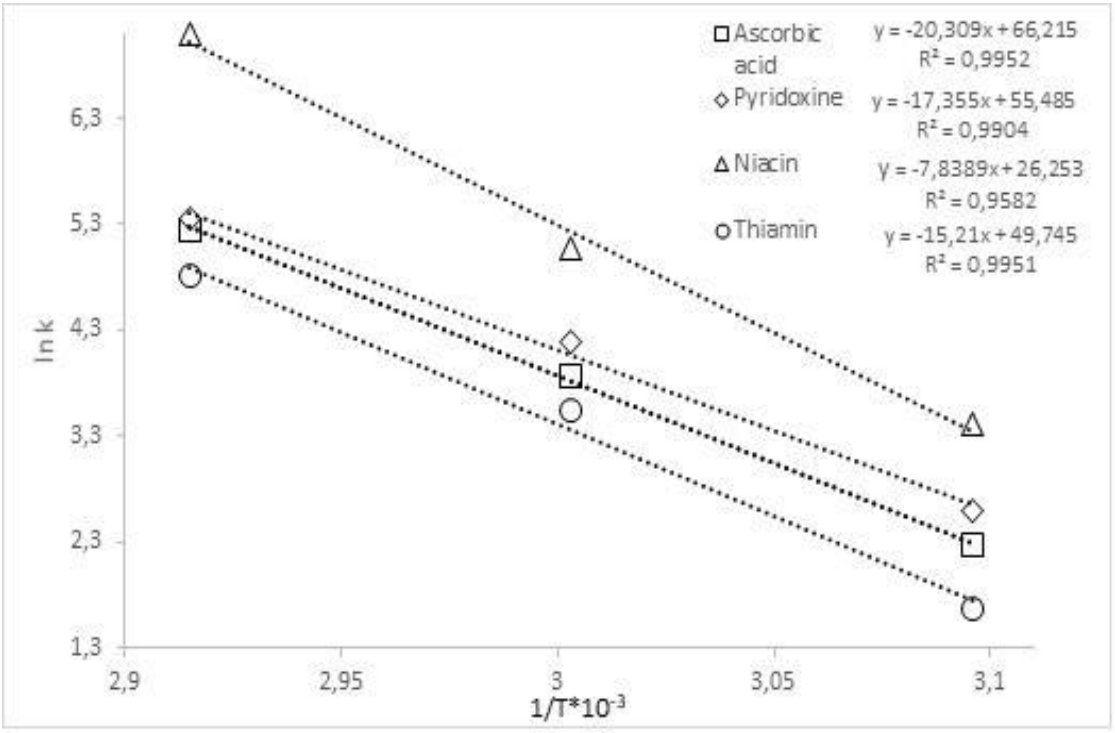
Ascorbic acid
y = -20,309x + 66,215
R² = 0,9952
Pyridoxine
y = -17,355x + 55,485
R² = 0,9904
Niacin
y = -7,8389x + 26,253
R² = 0,9582
Thiamin
y = -15,21x + 49,745
R² = 0,9951
ln k
6,3
5,3
4,3
3,3
2,3
1,3
2,9
2,95
3
3,05
3,1
1/T*10-3

Figure 5

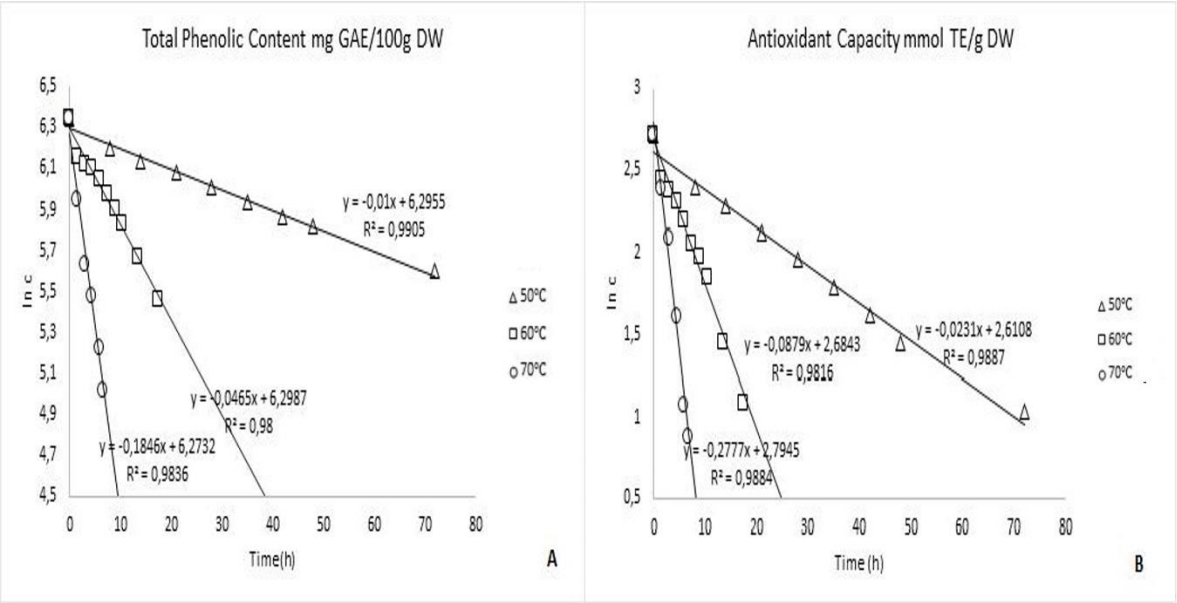
Total Phenolic Content mg GAE/100g DW
6,5
6,3
6,1
5,9
5,7
5,5
5,3
5,1
4,9
4,7
4,5
ln c
y = -0,01x + 6,2955
R² = 0,9905
y = -0,0465x + 6,2987
R² = 0,98
y = -0,1846x + 6,2732
R² = 0,9836
0 10 20 30 40 50 60 70 80
Time(h)
50°C
60°C
70°C
A
Antioxidant Capacity mmol TE/g DW
3
2,5
2
1,5
1
0,5
ln c
y = -0,0231x + 2,6108
R² = 0,9887
y = -0,0879x + 2,6843
R² = 0,9816
y = -0,2777x + 2,7945
R² = 0,9884
0 10 20 30 40 50 60 70 80
Time (h)
50°C
60°C
70°C
B

Figure 6

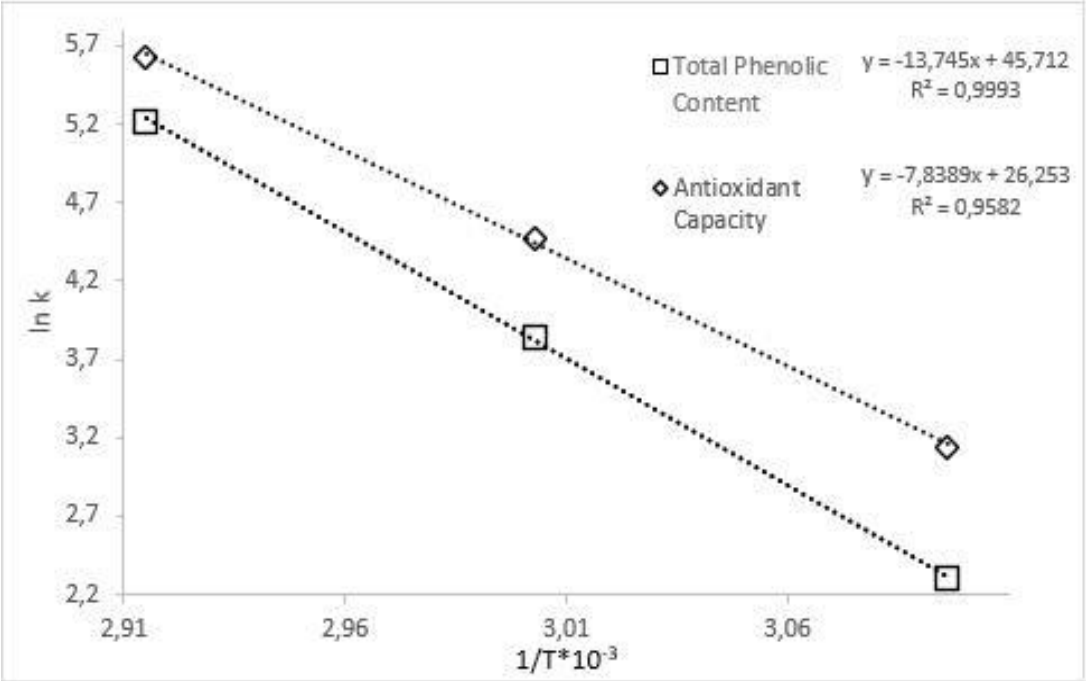
5,7
5,2
4,7
4,2
3,7
3,2
2,7
2,2
ln k
2,91
2,96
3,01
3,06
1/T*10-3
Total Phenolic Content
y = -13,745x + 45,712
R² = 0,9993
Antioxidant Capacity
y = -7,8389x + 26,253
R² = 0,9582

Figure 7