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Changes in Total Sugar, Ascorbic Acid, Alpha-Tocopherol and Beta-Carotene Contents of Rosehip Fruits Based on Harvest Times

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ABSTRACT

This study was conducted to determine the changes in total sugar, ascorbic acid, α-tocopherol and β-carotene content of fresh fruits of rosehip species during ripening, for three years. Fruits of selected genotypes belonging to *Rosa dumalis, R.canina* and *R.villosa* were used. Fruits were harvested in six different times based on hyphantium color change or flesh softening. The total sugars of *R.dumalis, R.canina,* and *R.villosa* fruits ranged between 11.22-16.68, 9.28-13.90, and 9.28-16.31%, respectively. Also, ascorbic acids varied between 767.1-1324.9, 168.7-481.5 and 241.2-574.2 mg 100 g^{-1}, respectively; α-tocopherols ranged between 5.87-10.25, 5.29-10.04 and 2.74-11.65 mg 100 g^{-1}, and β-carotenes varied between 0.18-2.03, 0.34-2.42 and 0.49-3.62 mg 100 g^{-1}, respectively. Total sugar, ascorbic acid, α-tocopherol and β-carotene content linearly increased depending on ripening time. Relationships between total sugar and vitamins were significant. It was concluded that harvest should be delayed as much as possible in order to obtain fruits with high content of total sugar and the main components of vitamins.

Keywords*:* Hips, Wild roses, Vitamin A, Vitamin C, Vitamin E.

INTRODUCTION

The fruits of the rosehip have been used in folk medicine for a long time, due to the prophylactic and therapeutic effects of rosehip against colds, infectious diseases, gastrointestinal disorders, urinary tract and inflammatory diseases (Ouerghemmi *et al*., 2016; Zahara *et al*., 2020).

In recent years, studies have proven that rosehip products are functional foods (Jiménez *et al*., 2017; Paunovic *et al*., 2019; Fascellaa *et al*., 2019). This character makes rosehip products much more remarkable and valuable. Rosehips differ from other fruits with their high vitamin C, vitamin E, phenolic, and antioxidant content, making it an economical source of antioxidants. (Kayahan *et al*., 2022). Rosehip fruits also contain a considerable amount of total soluble solids, sugars, vitamins, carotenoids, phenolics, and other antioxidant efficient compounds (Tumbas *et al*., 2012; Elmastas *et al*., 2017). In recent decades, rosehip fruit has been increasingly studied for its medical properties. It has been reported that rosehips contain several biologically active compounds such as flavonoids, tannins, carotenoids, fatty acids and vitamins, particularly vitamin C, E and provitamin A (Al-Yafeai *et al*. 2018B).

Sugars, synthesized during the fruit growth and ripening period, are both a harvest criterion for various fruit species and play an essential role in food processing or preserving. The sugar content of rosehip fruits rises with ripening like many other fruit species (Moirangthem and Tucker, 2018). Various studies have been conducted to reveal the total sugar and its fractions of

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rosehips (Yoruk *et al*., 2008).

Vitamin C together with polyphenols strengthens the arteries, reduces the risk of cancer due to antioxidant activity, helps dietary iron function in the body, works in adrenaline synthesis, and lowers blood cholesterol (Kadakal *et al*., 2002). Ascorbate deficiency leads to scurvy disease in the nutritional regime. A critical preliminary sign of vitamin C deficiency is fatigue (Samur, 2008).

Compounds of tocols (tocopherol and tocotrienol) show vitamin E activity (Colombo, 2010). The most active is α-tocopherol. The daily intake of vitamin E is 8-10 mg for adults (Samur, 2008). Vitamin E is crucial for the nervous system's functions, muscles, endocrine glands such as the pituitary, surrenal and reproductive organs. Rosehip fruits contain different α-tocopherol contents at different maturities (Yoruk *et al*., 2008; Andersson *et al*., 2011, 2012; Bhave *et al*., 2017)

Beta-carotene content of *Rosa dumalis, Rosa dumalis* hybrid*, Rosa rubiginosa* and *Rosa spinosissima* ranged from 8 to 373 mg kg^{-1} (Bhave *et al*., 2017). Epidemiological studies have shown an inverse relationship between degenerative diseases caused by oxidative stress and carotenoid consumption (Paiva and Russell, 1999).

Rosehip fruit is not suitable for table (fresh) consumption. It is common to process rosehip fruits in fruit juice, jam, marmalade, food additive (to enrich the vitamin content of foods, to color, etc.). It is obvious that these products are of great importance to be harvested at the right time in order to have high quality and high nutritional value. Otherwise, it will not be possible to obtain a product that can be considered as a functional food.

Assessment of optimal harvest time is vital in order to obtain rosehips suitable for the desired processed product. Compositional changes in total sugar and vitamins of rosehip fruits during ripening have great importance. The easy dropping of the fruit from the tree is a clear indicator of the ripening phase for various fruit crops. However, the abscission layer is not formed in rosehip stalks of fruits during ripening. Therefore, unpicked fruits usually remain on the branches until the following season. In this case, the reasonable or practical way to predict by the farmer that rosehip fruits reach harvest maturity may be to observe its color and fruit flesh firmness changes visually. Fruit color is also an indicator of the appearance and determination of fruit maturity in various fruit crops (Uggla *et al*., 2005; Andersson *et al*., 2011, 2012).

The contents of the over-ripe rosehip fruits and relationships between total sugar, ascorbic acid, α-tocopherol, and β-carotene contents of rosehip fruits have not been sufficiently investigated. Therefore, this study aimed to determine the optimum harvest times of *R.dumalis*, *R.canina,* and *R.villosa* hips depending on fruit color change, and to evaluate total sugar, ascorbic acid, α-tocopherol, and β-carotene changes during ripening.

MATERIALS AND METHODS

Chemicals

HPLC grade α-tocopherol and β-carotene were purchased from the Sigma-Aldrich Company (Sternheim, Germany). Liquid nitrogen, ethanol, hydrochloric acid, antron/sulfuric acid, oxalic acid, 2.6-dichlorophenolindophenol, hexane, ethyl alcohol for spektrofotometer were purchased from Merck Company (Istanbul, Turkey).

Plant Materials

The hips of selected genotypes of *R.dumalis, R.canina,* and *R.villosa,* located in the Garden of the Agricultural Research and Application Center of Gaziosmanpasa University, Tokat/Turkey, were used in the research in 2010, 2011, and 2012. The hips were harvested at six different times from the middle of the July to the end of the September. Hip color change was used as

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the basis for determining the harvest times of the first four harvests, whereas the softening of the hypanthium was used as the basis for the other two harvest times.

According to these criteria, the first Harvest (H-1) was performed when the fruit color began to turn (from green to) yellow. The second Harvest (H-2) was performed when fruit orange color conversion reached 50%; and the third Harvest (H-3) was performed when the whole fruit turned to orange. The fourth Harvest (H-4) was performed when the fruit was dark orange or bright red depending on the species. The fifth Harvest (H-5) was done when the fruit flesh started to soften, and the sixth Harvest (H-6) was performed when hypanthium was softened on a large scale (over-ripe) (Figure 1).

Approximately 500 g of fresh fruit were harvested from the rosehip bushes at each harvest time. Randomly, 25 g of fruit from these samples were taken for analysis, which were made with 3 replicates. Seeds, pappies, stalks, and calyx of the fruits were removed following the harvest and fruit's flesh was stored at 20°C in the deep freezer until analysis.

When the meteorological records of the Tokat Region (Figure 2) were examined, it was determined that the data for 2010 and 2012 were more similar during the harvest period.

Total Sugar Analysis (%)

A 25 g of fruit sample was crushed in a coffee grinder with liquid nitrogen. Then, 0.5 g was weighed, and 5 mL of ethanol (80%) extraction liquid was added to the fruit samples, vortexed, and centrifuged. The centrifuged samples were filtered, and 5 mL of ethanol was added to the remaining pulp, vortexed and centrifuged. The centrifuged samples were filtered, and the combined samples obtained were used in the analysis of water-soluble sugar. Hydrochloric acid (1.1%, 5 mL) was added to the remaining pulp, vortexed, and kept in boiling water for half an hour. These samples were centrifuged and evaluated in the analysis of water-insoluble sugar. An aliquot of 10 µL of the prepared samples were taken, and 990 µL of distilled water was added. After this dilution, 3 mL of antron/sulfuric acid (mg mL^{-1}) solution was added. The samples were vortexed after cooling in cold water and read at a wavelength of 620 nm in the spectrophotometer (UV-VIS Hitachi U-290, Japan). The total sugar was determined by adding the results obtained by replacing the values obtained from the readings in the calibration chart. The results were expressed as a percentage using an external standard calibration (Femenia *et al*., 1995).

Ascobic Acid Analysis [mg 100 g^{-1} Fresh Weight (FW)]

Ascorbic acid of rosehip fruits was determined by Khan *et al*. (2006) spectrophotometric method. A 5 g sample of fruit was homogenized in 50 mL of distilled water and 10 mL of these samples were taken and centrifuged at 4,000 rpm for 5 minutes, then, the samples were taken from the upper part of the supernatant for analysis. An aliquot of 100 µL of this prepared extract was taken, then, 400 µL of 0.4% oxalic acid and 4.5 mL (30 ppm) 2.6-dichlorophenolindophenol solution were added. The mixture was vortexed and read immediately on the 520 nm wavelength spectrophotometer. The amount of ascorbic acid in the fruit was calculated using the calibration chart. Ascorbic acid was used to prepare the calibration chart.

α-Tocopherol and β-Carotene Analysis

The modified extraction method developed by Kazaz *et al*. (2009) was used to determine *α*-tocopherol. Fruit samples (25 g) were ground in a coffee grinder with liquid nitrogen. Powdered fruit sample (2 g) was taken, and 5 mL of n-hexane was added and vortexed for 2 minutes. A 1 mL sample

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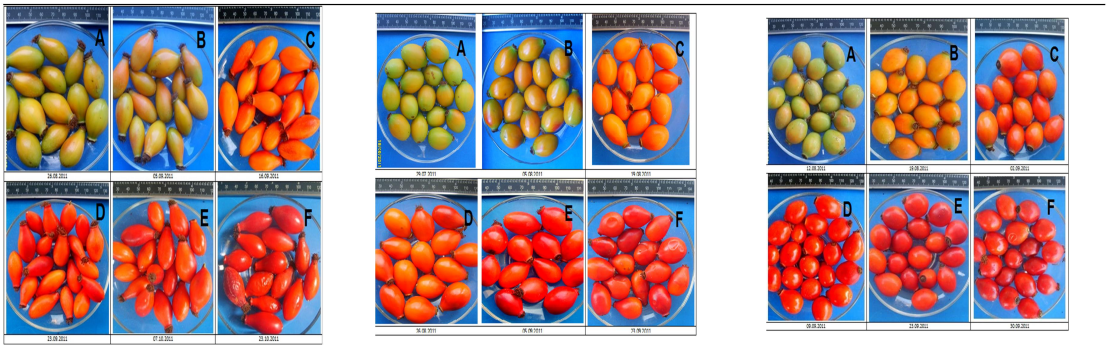

Figure 1. Fruit color stages of species[x] (A: Harvest-1, B: Harvest-2, C: Harvest-3, D: Harvest-4, E: Harvest-5, F: Harvest-6).

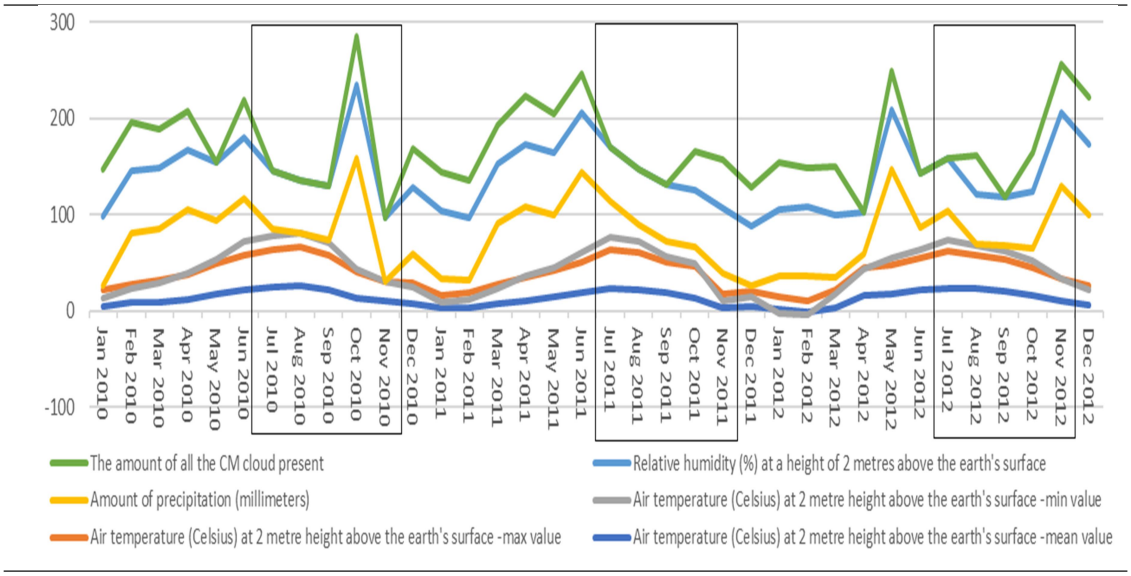

Figure 2. Meteorological records of the administrative central of Tokat during the study years (Anonymous, 2023).

was taken from the obtained supernatant, and its solvent was removed and poured in tubes. It was stored in the dark in the refrigerator until HPLC analysis. Before HPLC analysis, it was re-dissolved with ethyl alcohol, filtered through micro-filters. The amount of α-tocopherol and β-carotene in the samples was calculated using the calibration curve constructed using the standards, and the results were presented as mg 100 g^{-1}. Extraction prepared for α-tocopherol analysis was also used for β-carotene.

Qualitative analysis of α-tocopherol and β-carotene were performed in the HPLC system consisting of Shimadzu brand LC 20AT pump and SPD-M20A model DAD detector and CTO-20AC (Japan) model column furnace. In the analysis and chromatographic separations of standards, Wakosil (150×4.60 mm, 5 µm) was made with the C18 reverse-phase filling column. The mobile phase was set to 100% methanol, and the mobile phase flow rate to 1 mL min^{-1}. Samples and standards device was injected as 20 µL, and the column temperature was adjusted to 50°C. The

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reading was done at a wavelength of 295 nm for α-tocopherol and 450 nm for β-carotene.

Statistical Analysis

All statistical analyses were performed using the SPSS (15.0 version). Three rosehip bushes were selected for each species and samples were taken for three replicates in each harvest time. Data were subjected to Analyses Of Variance (two-way ANOVA) (Table 1) followed by Duncan test of *Rosa* species, and obtained the values at harvest times. In addition, an evaluation was made among years, species, harvests and their interactions. Besides, a Pearson test was performed on the correlations of the obtained data and the results were given. In order to determine the relationship among the three years averages, graphics were created (Excel 365) and R^2 value was calculated.

RESULTS AND DISCUSSION

The total sugar content of fresh rosehip fruits is important in determining the procedures to be applied at the processing stage, determining the products' energy content, and creating a standard product. High total sugar content of industrial products also reduces the amount of external sugar addition. In our investigation, the change in total sugar content between years, harvests and species was found significant (Table 1). Significant increases were also observed from the first harvest to the last one in all the species (Tables 2, 3, and 4). The total sugar changed between 11.22-16.68% (*R. dumalis*), 9.28-13.90% (*R. canina*), and 9.28-16.31% (*R. villosa*). The total sugars of species have increased linearly (Figure 3) during ripening, and the total sugar amounts of *R. dumalis* and *R. villosa* was higher than those of *R. canina*. Unlike the first and third year, total sugar rate was found to be lower in all three species in the second year. It is estimated that this situation is due to the low mean temperature and rainfall values in the same year (Figure 2). Uggla *et al*. (2005) determined the total sugar between 131.0-227.5 mg g^{-1} DW in *R. dumalis* and 28.6-155.2 mg g^{-1} DW in *R. rubiginosa* fruits. These researchers noted that the total sugar increased during ripening, but some variations occurred in the last two harvests. Yoruk *et al*. (2008) indicated the total sugar content of fresh fruit as 24.70 mg g^{-1} in *R. villosa*, 30.72 mg g^{-1} in *R. canina*, and 30.97 mg g^{-1} in *R. dumalis*. Rosu *et al*. (2011) observed that total sugar content of 10 *Rosa* species grown in the northeastern region of Romania were 12.05-17.63 g 100 g^{-1} FW. Barros *et al*. (2011) found the total sugar as 7.25 g 100 g^{-1} in unripe and 20.46 g 100 g^{-1} in ripe *R. canina* fruits. Barros *et al*. (2010) and Ozrenk *et al*. (2012) recorded total sugar as 26.90 g 100 g^{-1} and 16.46-27.01 g 100 g^{-1}, respectively. Kayahan *et al*. (2022) reported the amount of total sugar (average of two years) of *R. corymbifera, R. rugosa, R. alba,* and *R. canina* as 5.36, 2.10, 4.99 and 4.99%, respectively. Total sugar is expected to increase regularly with maturation. However, various results obtained by previous researchers have revealed that it can vary depending on variables such as ecological conditions, rosehip species, and harvest time.

Among the horticultural crops, rosehips fruits are known as the essential crops in terms of vitamin C. So, it is crucial to harvest the rosehip fruit at the right time when the vitamin C content is the maximum. The variations of vitamin C contents have been significant depending on year, harvest time, and species (Table 1). Vitamin C contents of our fresh fruits harvested from *R.dumalis, R.canina,* and *R.villosa* bushes varied between 767.17-1325.92, 168.75-481.59, and 241.24-574.29 mg 100 g^{-1} values depending on ripening, respectively (Tables 2, 3, and 4; Figure 4). The differences between the harvest times in *R.dumalis, R.canina,* and *R.villosa* were significant, considering the average of three years of data. However, when the years were evaluated separately, there was no significant difference between the fifth and the sixth harvest time. Vitamin C

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Table 1. Mean squares and significances from analysis of total sugar, ascorbic acid, α-tocopherol and β-carotene content of Rosa species sampled on different harvesting dates during three years. [a]

Source	DF	Ascorbic acid Mean Square	Sig.	Total sugar Mean square	Sig.	β-Carotene Mean square	Sig.	α-Tocopherol Mean square	Sig.
Year (Y)	2	1198803.273	0.000	100.319	0.000	6.890	0.000	951.215	0.000
Species (S)	2	8646453.693	0.000	68.941	0.000	3.657	0.000	29.082	0.014
Harvest (H)	5	499728.701	0.000	110.434	0.000	13.939	0.000	68.111	0.000
Y×S	4	508617.521	0.000	7.263	0.031	0.215	0.526	24.704	0.007
Y×H	9	116108.944	0.000	4.237	0.121	1.113	0.000	27.970	0.000
S×H	10	40728.259	0.000	2.104	0.627	0.841	0.002	20.441	0.002
Y×S×H	18	42064.293	0.000	6.802	0.001	0.265	0.477	13.128	0.015
Error	102	3582.797		2.624		0.268		6.526	

[a] DF: Degrees of Freedom.

Table 2. Changes in total sugar, α-tocopherol, β-carotene and ascorbic acid content of *R. dumalis* hips during ripening of three years.[a]

Harvest	2010	2011	2012	Average
		Total sugar (%)		
H-1	13.39a	7.25c	13.01b	11.22b
H-2	13.24a	8.18c	14.21b	11.88b
H-3	14.52a	11.17b	14.31b	13.33ab
H-4	14.65a	13.49ab	14.37b	14.17ab
H-5	14.00a	13.67ab	17.10a	14.92ab
H-6	--	15.88a	17.48a	16.68a
		α-Tocopherol (mg 100 g^{-1})		
H-1	9.72a	5.33d	2.57c	5.87a
H-2	6.66ab	15.34ab	4.62abc	8.87a
H-3	8.45ab	10.24bcd	4,23bc	7.64a
H-4	5.84 b	18.47a	6.45ab	10.25a
H-5	7.03ab	11.64bc	7.57a	8.75a
H-6	--	8.03cd	5.99ab	7.01a
		β-Carotene (mg 100 g^{-1})		
H-1	0.12a	0.16c	0.25d	0.18c
H-2	0.11a	0.46c	0.56d	0.38bc
H-3	0.31a	0.62c	1.02cd	0.65abc
H-4	0.49a	2.97a	1.43bc	1.63ab
H-5	0.36a	1.74b	2.10ab	1.40ab
H-6	--	1.74b	2.31a	2.03a
		Ascorbic acid (mg 100 g^{-1})		
H-1	839.60b	629.00b	832.92d	767.17c
H-2	984.60ab	713.17a	1030.83c	909.53b
H-3	1065.93ab	746.50a	1235.00b	1015.81b
H-4	1075.60ab	732.33a	1362.08b	1056.67b
H-5	1212.93a	713.17a	1951.67a	1292.59a
H-6	--	727.33a	1922.50a	1324.92a

[a] (a-d): The difference between the means indicated by different letters in the same column. ($P< 0.05$) is significant. H: Harvest.

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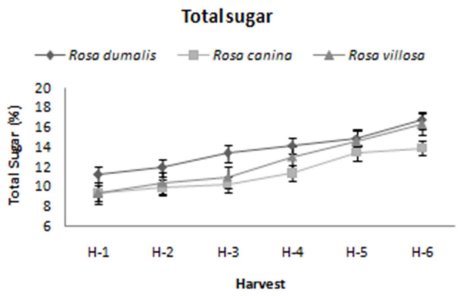

Figure 3. Change in total sugars content depending on the ripening in rosehip species (Average of 3 years).

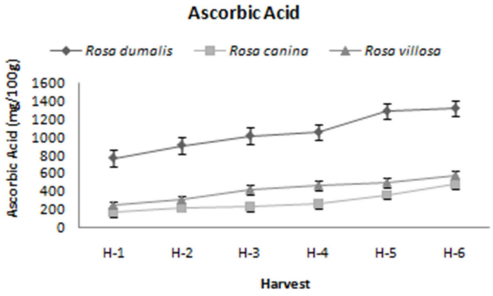

Figure 4. Change in A. acid content depending on the ripening in rosehip species (Average of 3 years).

content of rosehip fruits of some published researches was reported as between 108.57-908.57 mg 100 g^{-1} (Gunes and Dolek, 2010), 27.49 mg 100 g^{-1} (Egea *et al*., 2010), 614.45-866.91 mg 100 g^{-1} (Rosu *et al*., 2011), 3–24 µg gr^{-1} dry matter (Jiménez *et al*., 2017) and 385,82–736,27 mg 100 g^{-1} (Medveckienė *et al*., 2020). In a study conducted on green, orange, and red maturation periods of *Rosa rugosa*, the amount of ascorbic acid was determined as 955, 1,090 and 798 mg 100 g^{-1}, respectively (Al-Yafeai *et al*., 2018, A). The amount of ascorbic acid (average of two years) of *R. corymbifera, R. rugosa, R. alba,* and *R. canina* were found to be 830, 575, 816 and 695 mg 100 g^{-1}, respectively (Kayahan *et al*., 2022). Our and previous studies' results demonstrated a high variation in vitamin C contents based on the species and the harvesting time. In this context, results of vitamin C content of the current study are compatible with the literature. Even the results obtained by Nojavan *et al*. (2008) showed that the amount of vitamin C in fully ripe fruits of rosehip was about six times more than that in the orange or half-ripe time. However, no literature has shown vitamin C content of overripe or soften rose hips. Contrary to our expectation, vitamin C was also high in the last two harvests, and this means that the synthesis of vitamin C in rosehips continues until over-ripe. However, these findings need to be supported by further studies.

Alpha-tocopherol values of *R. dumalis, R. canina,* and *R. villosa* varied between 5.87-10.25, 5.29-10.04, and 2.74-11.65 mg 100 g^{-1}, respectively (Tables 2, 3, 4). Significant differences have been determined when α-tocopherol contents of rosehip fruits are examined based on year, species, and harvest time individually (Tables 1, 2, 3, 4). However, it is more important to consider the average of years. In this case, a regular and non-significant increase occurred between harvest times during ripening, except for *R. dumalis* (Figure 5). Following the fourth harvest, α-tocopherol decreased in *R. dumalis*, but the difference between the harvests was not statistically significant, depending on the average of three years (Table 2). Various results were obtained from published studies based on α-tocopherol. As in some previous studies, α-tocopherols were determined as 3.57-17.60 µg g^{-1} (Yoruk *et al*., 2008), 21.62 µg g^{-1} (Kazaz *et al*., 2009), 7.05 mg 100 g^{-1} Barros *et al*., 2010), 15 and 245 mg mg^{-1}, and (Bhave *et al*., 2017). Barros *et al*. (2011) reported that the mean α-tocopherol of unripe and ripe fruit of *R. canina* ranged between 9.93 and 52.13 mg 100 g^{-1}. Andersson *et al*. (2012) reported that α-tocopherol changes were detected during ripening in rosehip, based on fourteen harvest times. Alpha-tocopherols were determined in the *R. rubiginosa, R. dumalis, R. dumalis hybrida,* and *R.spinosissima* as 126.9-169.5, 97.3-121.0, 121.7-147.9 and 153.6-232.1 µg g^{-1} DW, respectively. In the study conducted on *Rosa rugosa* in green, orange, and red maturation periods, the amount of α-tocopherol were determined as 15, 17 and 14 µmol 100 g^{-1}, respectively

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Table 3. Changes in total sugar, α-tocopherol, β-carotene and ascorbic acid content of *R. canina* hips during three of years ripening.[a]

Harvest	2010	2011	2012	Average
		Total sugar (%)		
H-1	9.43b	8.39bc	10.03c	9.28c
H-2	9.90b	7.67bc	12.16b	9.91bc
H-3	11.19ab	6.99c	12.55ab	10.24bc
H-4	12.24ab	9.39b	12.55ab	11.39abc
H-5	14.04a	12.59a	13.70ab	13.44ab
H-6	--	13.79a	14.01a	13.90a
		α-Tocopherol (mg 100 g^{-1})		
H-1	4.00c	9.57a	2.29ab	5.29a
H-2	6.10ab	10.85a	1.10b	6.02a
H-3	7.02a	9.82a	1.51b	6.12a
H-4	4.85bc	15.13a	2.28ab	7.42a
H-5	6.56ab	16.12a	2.27ab	8.32a
H-6	--	16.25a	3.83a	10.04a
		β-Carotene (mg 100 g^{-1})		
H-1	0.09b	0.35c	0.59c	0.34c
H-2	0.33ab	0.48c	0.61c	0.47c
H-3	0.40a	0.38c	0.77c	0.52c
H-4	0.50a	1.07bc	1.10bc	0.89bc
H-5	0.51a	1.66ab	1.58b	1.25b
H-6	--	2.31a	2.53a	2.42a
		Ascorbic acid (mg 100 g^{-1})		
H-1	184.33a	172.33b	149.58e	168.75c
H-2	206.42a	198.17b	251.67d	218.75bc
H-3	212.04a	200.67b	280.83d	231.18bc
H-4	181.21a	244.00b	368.33c	264.51bc
H-5	205.58a	365.67a	518.33b	363.19ab
H-6	--	378.17a	585.00a	481.59a

[a] (a-e): The difference between the means indicated by different letters in the same column. ($P< 0.05$) is significant. H: Harvest

(Al-Yafeai *et al*., 2018A). Kayahan *et al*. (2022) reported the average amount of α-tocopherol of *R. corymbifera, R. rugosa, R. alba,* and *R. canina* as 8.03, 10.07, 9.38 and 10.36 µmol 100 g^{-1}, respectively. These researchers reported that the mean α-tocopherol was generally higher for early harvesting dates than for late harvesting. However, the differences in tocopherol content during the season were relatively small, and the most considerable difference was found for α-tocopherol. Results obtained on α-tocopherols from our study confirmed the results obtained by Andersson *et al*. (2012), but lower than those of Barros *et al*. (2011).

The change in β-carotene content between years, harvests, and species were found to be statistically significant (Table 1). Beta-carotene values obtained from *R. dumalis, R. canina,* and *R. villosa* , depending on harvest times, ranged between 0.18-2.03, 0.34-2.42, and 0.49-3.62 mg 100 g^{-1}, respectively (Tables 2, 3, 4). When three years were analyzed collectively or separately, it was seen that the sixth time was the most appropriate for harvesting (Figure 6). Our values obtained from the harvest times in different ripening were higher than those of

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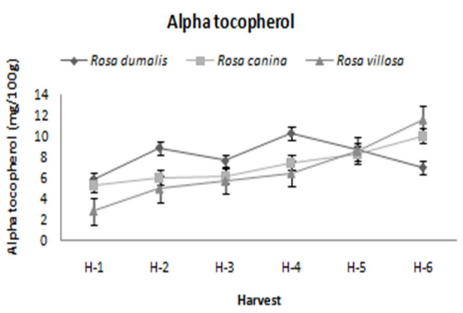

Figure 5. Change in α-tocopherol content depending on the ripening in rosehip species (Average of 3 years).

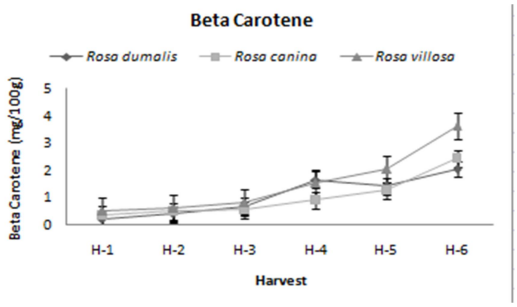

Figure 6. Change in β-carotene content depending on the ripening in rosehip species (Average of 3 years).

Table 4. Changes in total sugar, α-tocopherol β-carotene and ascorbic acid content of *R. villosa* hips during ripening of three years. [a]

Harvest	2010	2011	2012	Average
		Total sugar (%)		
H-1	10.32bc	8.11 b	9.40b	9.28c
H-2	11.42 b	9.16 b	10.56b	10.38bc
H-3	08.68 c	12.47ab	11.72b	10.96bc
H-4	11.59 b	11.69ab	15.68a	12.99ab
H-5	14.16 a	12.53ab	16.84a	14.51a
H-6	--	15.63 a	16.98a	16.31a
		α-Tocopherol (mg 100 g^{-1})		
H-1	2.55b	4.61c	1.06d	2.74d
H-2	5.10ab	8.03bc	1.53d	4.89c
H-3	6.78ab	8.27bc	1.96cd	5.67c
H-4	5.28ab	11.42abc	2.52bc	6.41bc
H-5	7.86a	14.68ab	3.23b	8.59b
H-6	--	17.30a	5.99a	11.65a
		β-Carotene (mg 100 g^{-1})		
H-1	0.29a	0.30c	0.87d	0.49c
H-2	0.41a	0.69c	0.65d	0.58c
H-3	0.61a	0.72c	1.01cd	0.78c
H-4	0.97a	1.93bc	1.67c	1.52bc
H-5	0.83a	2.77ab	2.42b	2.01b
H-6	--	3.85a	3.38a	3.62a
		Ascorbic acid (mg 100 g^{-1})		
H-1	306.39c	199.00c	218.33d	241.24c
H-2	373.89b	203.17c	337.08c	304.71bc
H-3	420.00ab	394.00b	439.17b	417.72ab
H-4	377.78b	439.00b	576.67a	464.48ab
H-5	433.61a	417.33b	637.08a	496.01a
H-6	--	507.33a	641.25a	574.29a

[a] (a-d): The difference between the means indicated by different letters in the same column. ($P< 0.05$) is significant. H: Harvest

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Table 5. Correlation between average ascorbic acid, total sugar, β-carotene, α-tocopherol content, harvests, years and species of all samples of *Rosa* species harvested at different harvest dates in three years (n= 153).

Character	Ascorbic acid	Total Sugar	β-Carotene	α-Tocopherol	Harvest	Year	Species
Ascorbic acid	1						
Total Sugar	0.567**	1					
β-Carotene	0.204*	0.517**	1				
α-Tocopherol	0.023	0.016	0.447**	1			
Harvest	0.308**	0.627**	0.692**	0.302**	1		
Year	0.206*	0.221**	0.370**	-0.266**	0.118	1	
Species	-0.394**	-0.079	0.195*	-0.127	0	0	1

* Correlation is significant at the 0.05 level; ** Correlation is significant at the 0.01 level.

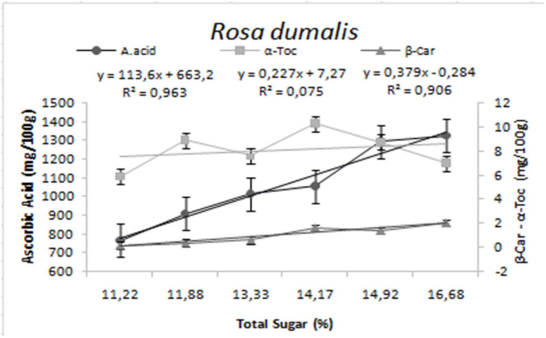

Figure 7. Correlation between total sugars and vitamins in *R. dumalis* hips (Average of 3 years).

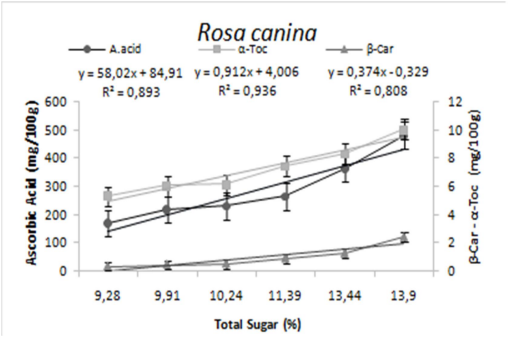

Figure 8. Correlation between total sugars and vitamins in *R. canina* hips (Average of 3 years).

Kazaz *et al*. (2009) (3.25 µg g^{-1}) and were lower than Egea *et al*. (2010) (18.07 mg 100 g^{-1}), Rosu *et al*. (2011) (24.64 -34.32 mg 100 g^{-1}), Barros *et al*. (2011) (25.88- 97.77 mg 100 g^{-1}), Bhave *et al*. (2017) (8 to 373 mg kg^{-1},) and Medveckienė *et al*. (2020) (3.95 – 31.40 mg 100 g^{-1}), but were similar to Barros *et al*. (2010) (1.29 mg 100 g^{-1}) and Andersson *et al*. (2011) (18.70-196.66 µg g^{-1} DW). According to Andersson *et al*. (2011), β-carotene increased from the first harvesting date (Aug 4) to later harvesting dates (Oct 12, Oct 19) and decreased irregularly in the last three weeks. Al-Yafeai *et al*. (2018A) reported the β-carotene content of *Rosa rugosa* in green, orange, and red maturation stages as 1.8, 7.0 and 7.3 mg 100 g^{-1}, respectively. As can be seen, different results have been obtained by different researchers. Investigating the internal and external factors affecting the synthesis of vitamins and their relationships under controlled conditions may allow more accurate evaluations.

The total sugar content of rosehip species and the relationships between ascorbic acid, α-tocopherol and β-carotene were examined. Except for α-tocopherol (R^2= 0.07) belonging to *R. dumalis*, a very high (R^2= 0.81-0.97) positive relationship was found between total sugar and ascorbic acid, α-tocopherol and β-carotene (Figures 7, 8, 9). While all the bioactive compounds analyzed in *R. canina* and *R. villosa* showed a steady increase due to maturation, this situation was not observed in *R. dumalis.*

Mean ascorbic acid content of *R. dumalis* fruits showed considerable increase after the fourth Harvest time (H-4), and its relationship with total sugar was found very high (R^2= 0.96). Alpha-tocopherol content exhibited a rapid decrease after the fourth

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Table 6. Correlation between average ascorbic acid, total sugar, β-carotene and α-tocopherol content of *Rosa* species sampled on different harvesting dates in three years (n= 51).

Rosa dumalis[a]				
	AA	TS	BC	AT
AA	1			
TS	0.641**	1		
BC	0.346*	0.430**	1	
AT	-0.313*	-0180	0.458**	1
Rosa canina[a]				
	AA	TS	BC	AT
AA	1			
TS	0.568**	1		
BC	0.758**	0.477**	1	
AT	-0.055	-0.207	0.345*	1
Rosa villosa[a]				
	AA	TS	BC	AT
AA	1			
TS	0.736**	1		
BC	0.608**	0.686**	1	
AT	0.130	0.280*	0.620**	1

[a] AA: Ascorbic Acid, TS: Total Sugar, BC: Beta-Carotene, AT: Alpha-Tocopherol.
* Correlation is significant at the 0.05 level
** Correlation is significant at the 0.01 level

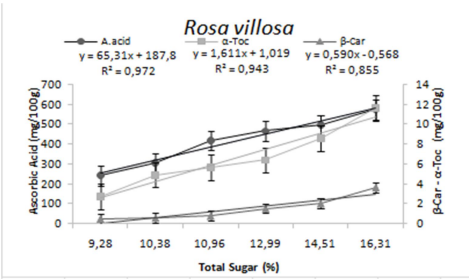

Figure 9. Correlation between total sugars and vitamins in *R. villosa* hips (Average of 3 years).

Harvest (H-4), and its relationship with total sugar was low (R^2= 0.07). Beta-carotenes content was lower than the other two compounds and increased until the fourth Harvest time (H-4), and its relation with total sugar was high (R^2= 0.90) (Figure 7).The ascorbic acid content of *R. canina* also increased rapidly after the fourth Harvest time (H-4), and its relationship with total sugar was high (R^2= 0.89). Alpha-tocopherol content demonstrated no significant increase after the third Harvest time (H-3), and its relationship with total sugar was found high (R^2= 0.93). Beta-carotene content was lower than the other two compounds, and its relationship with total sugar was high (R^2= 0.80) (Figure 8). The Ascorbic acid content of *R. villosa* exhibited a regular increase from the first to the last harvest time, and its relationship with total sugar was very high (R^2= 0.97). Alpha-tocopherol also increased significantly after the fourth Harvest time (H-4), and its relationship with total sugar was high (R^2= 0.94). Beta-carotene content was lower in amount than the other two compounds, and its relationship with total sugar was high (R^2 =0.85) (Figure 9).

Significant differences were determined among harvest times, years, and species studied. Pearson correlation test results revealed strong interactions, except α-tocopherol when the examined traits were compared (Table 5). While there was a correlation between α-tocopherol and β-carotene, no correlation was determined between α-tocopherol and total sugar and α-tocopherol and ascorbic acid in all species (Table 6). In particular, total sugar and vitamin C varied similarly in 2010 and 2012. In fact, the similarity of the climate data (Figure 2) of both years supports this situation. It is possible that there is a correlation between the two features and climatic conditions. However, a more detailed and comprehensive study is needed to use precise expressions. Thus, it is important to determine the harvest time correctly depending on the ecological conditions, year, and species (Figure 2).

CONCLUSIONS

The total sugar content of *R.dumalis, R.canina* and *R.villosa* rose hips ranged between 11.22-16.68, 9.28-13.90 and 9.28-

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16.31%, respectively. Ascorbic acids were found between 767.17-1324.92, 168.75-481.59 and 241.24-574.29 mg 100 g^{-1}; α-tocopherols were between 5.87-10.25, 5.29-10.04 and 2.74-11.65 mg 100 g^{-1} and β-carotenes were recorded as 0.18-2.03, 0.34-2.42 and 0.49-3.62 mg 100 g^{-1}, respectively. As a result, when the values were evaluated individually based on year or as the averages of the years of cultivation, total sugar, ascorbic acid, α-tocopherol, and β-carotene increased during ripening. While all bioactive compounds measured in *R. canina* and *R. villosa* hips exhibited a linear increase depend on ripening, *R. dumalis* showed irregularities towards the end of late harvest (Figures 3-6). Although it is possible to talk about the effect of ecological conditions in general, detailed studies are needed under controlled conditions for new products such as rose hips. *R.dumalis* stood out with its high ascorbic acid, α-tocopherol, and β-carotene values. The synthesis of vitamin C of rosehips continued until over-ripe. These promising results seem to suggest that *R. dumalis* could be used in large-scale production of hips, or in breeding studies. Delaying the harvest caused higher content of these bioactive substances in the rosehip fruits. However, rosehip fruits must be harvested before they soften in order to avoid smashing during harvest and following processes and to preserve their integrity. Late harvesting is recommended if high content of carotenoids is desired, while harvesting should be carried out earlier if a higher vitamin E and vitamin C content is desired, which in turn affects the antioxidants capacity (Al-Yafeai *et al*., 2018A). It was shown that the fifth harvest time was the correct or optimum time for species. However, considering the problems caused by over-ripening, it was concluded that rosehip fruits should be harvested just before softening, i.e., at the end of the fourth harvest time or at the beginning of the fifth harvest time. Although it is a desired feature of the fruit to be bigger, it is more important to have high phytochemical content due to its processing in the food industry as a functional food.

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تغییرات قند کل، اسید اسکوربیک، آلفا توکوفرول و بتاکاروتن میوه های گل سرخ بر اساس زمان برداشت

م. گونز، و ی. دولک

چکیده

این پژوهش به منظور تعیین تغییرات قند کل، اسید آسکوربیک، آلفا توکوفرول و بتاکاروتن در میوه‌های (rosehip) تازه گونه‌های گل سرخ در طی مرحله رسیدن به مدت سه سال انجام شد. از میوه هاي منتخب ژنوتیپ هاي *Rosa dumalis*، *R.canina* و *R.villosa* استفاده شد. میوه ها در شش زمان مختلف بر اساس تغییر رنگ هیفانتیوم(hyphantium) یا نرم شدن گوشت برداشت شدند. کل قندهای میوه های *R.dumalis*، *R.canina* و *R.villosa* به ترتیب بین ۱۶.۶۸-۱۱.۲۲% -۱۳.۹۰- ۹.۲۸% و -۱۶.۳۱- ۹.۲۸% بود. همچنین اسیدهای آسکوربیک به ترتیب بین ۱۳۲۴.۹-۷۶۷.۱، ۴۸۱.۵ -۱۶۸.۷و ۵۷۴.۲- ۲۴۱.۲ میلی گرم در ۱۰۰ گرم متغیر بودند. α-توکوفرول ها بین۱۰.۲۵ - ۵.۸۷، ۵.۲۹ - ۱۰.۰۴ و ۱۱.۶۵- ۲.۷۴ میلی گرم در ۱۰۰ گرم، و β-کاروتن ها بر حسب میلی گرم در صد گرم به ترتیب بین ۲.۰۳- ۰.۱۸، -۲.۴۲ -۰.۳۴ و -۳.۶۲ - ۰.۴۹ متغیر بودند. میزان قند کل، اسید اسکوربیک، آلفا توکوفرول و بتاکاروتن بسته به زمان رسیدن به صورت خطی افزایش یافت. ارتباط بین قند کل و ویتامین ها معنی دار بود. نتیجه گیری شد که برداشت باید تا حد امکان به تعویق بیفتد تا میوه هایی با محتوای قند کل بالا و اجزای اصلی ویتامین ها بدست آید.

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