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Compositional variation of olive fruit during ripening

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Abstract

Compositional changes during olive fruit ripening were studied in two important olive varieties grown in Turkey. Amounts of total and individual sugars (glucose and fructose) in olive fruits were determined. Total oil, fatty acid composition and 14 minerals were detected in the cultivars of olive fruit during different ripening periods. The relationship between the oil accumulation and sugars in olive fruits has been evaluated. The oil accumulation increased during the period of ripening while the sugar accumulation decreased. © 2000 Elsevier Science Ltd. All rights reserved.

1. Introduction

The biochemical process of oil accumulation in the olive fruit and the precursors for its synthesis during the period of fruit maturation has received considerable attention in recent years. Acetyl Co-A is considered to be needed for fatty acid synthesis in the seeds. Carbohydrates serve as a source for the acetyl needed for fatty acid biosynthesis (Simcox, Garland, Deluca, Canvin & Dennis, 1979). Therefore, both total and individual sugars have been measured in olive fruits but only at the beginning and end of the fruit development period. Glucose, fructose and mannitol were found to be the predominant sugars in the olive fruit (Patumi, Fontanazzi, Baldoni & Brambilla, 1990). Wodner, Lavee and Epstain (1987) studied the oil and sugar content during development and maturation of the fruit in different cultivars. In the cultivar having the least amount of oil, there was a parallel rise in oil and sugar levels at the beginning of the fruit ripening period, while the sugar level decreased in other cultivars having a higher oil content.

On the other hand, oil accumulation in olive fruits cannot be based only on changes in the sugars and polyol composition (Wodner et al., 1987). It has been suggested that the oil accumulation depends more on the ability of different cultivars to metabolise substrates than on a deficit of the same compounds. The particular role of maleic and citric acids of olive fruit has been noted (Patumi et al., 1990).

Olive composition is influenced by environmental and cultivar differences. However, certain fatty acids and minor components determine the quality of the oil. The olive's composition in several cultivars and correlations of the levels of several elements with some quality parameters in olive has been studied (Jordao & Lietao, 1990). While there are extensive data on the composition of the olive fruit, there is limited information on the oil accumulation and the precursors for its synthesis and their correlation during the period of olive maturation. Thus, two varieties of olive (Memecik and Domat), which are the most common cultivars in the Egean Region of Turkey, were chosen in this study. Memecik variety alone constitutes more than 50% of olive production in that region (Mendilcioğlu, 1990). The purpose of this study was to investigate the changes of some compounds in olive fruit during the period of maturation: specifically, oil content, sugars, fatty acids, some minerals and the relationship between the sugars and oil content.

2. Materials and methods

2.1. Materials

The experiments were carried out on the fruits from four selected olive trees belonging to two varieties of Memecik and Domat cultivated in the orchard of the

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Olive Research Institute, Bornova, İzmir, Turkey. Sufficient amounts of olive fruit samples were hand-picked from all sides of olive trees once-a-month from early September to the beginning of February during 1997. The collected samples for each variety were placed polyethylene bags and maintained at −20°C until analysed.

2.2. Methods

The number of olive fruits in a 1000 g sample was determined according to the Turkish Standards (Anon., 1992). Average weight of olive fruit was determined by weighing. For flesh separation, fruits were cut in half horizontally with a stainless-steel knife and the stones were removed and weighed. The flesh content was calculated by subtracting the stone weight from the whole olive fruit weight. The flesh to stone weight ratio (F/S) was determined by dividing the flesh weight by the stone weight. Flesh samples were blended in a Waring mechanical blender (capacity of 1000 ml) and homogenised. Aliquots from this homogenate were used for analysis.

Moisture content was determined by oven-drying method at 105 ± 1°C (NÜVE FN 500 Model). Oil content was determined by Soxhlet extraction using *n*-hexane as described by IUPAC (1979) method 1.122. Fatty acid methyl esters were prepared by trans-esterification using a methylant solution consisting of benzene, methanol and concentrated sulphuric acid (25:75:1, v/v/v). Gas chromatography (GC) of methyl esters was performed on a Shimadzu GC apparatus (Model 14 B) equipped with a hydrogen flame ionization detector. Temperatures of injector, oven and detector were 275, 185 and 275°C, respectively. A glass column (200 cm×3.2 mm i.d.) containing 15% DEGS on 80–100 mesh chromosorb was used for fatty acid analysis. Nitrogen served as the carrier gas at a flow rate of 90 ml/min and injection quantity was 0.5 ml. Retention times and peak areas were automatically computed by the data processor (Shimadzu C-R4A Chrompack). Identification was accomplished by comparing the retention time of unknown methyl esters with those of known fatty acid methyl ester standards.

Total sugar was estimated by the Lane–Eynon volumetric method. For the analysis of individual sugars (glucose and fructose), 5 g of homogenised olive flesh sample was weighed and 50 ml of distilled water were added. The resulting flesh–water mixture was stirred with a magnetic stirrer in boiling water for 20 min at 60°C. The extract was cooled and then the volume was made up to 100 ml with distilled water, filtered through ordinary and Whatman No.42 filter paper, successively. Ten ml of this clear extract were taken and refiltered through an Alltech 0.45 μm filter and injected onto the chromatographic column [25 cm×4.6 mm o.d., Shimtpack CLC-H2(M) Shimadzu Model]. Elution was done with a mixture of acetonitrile and water (75:25, v/v) at a flow rate of 1.2 ml/min at ambient temperature (25°C). The eluent was monitored by refractive index detector. Injection volume was 20 μl. The height of recorded peaks for each sample was compared with heights of peaks from a standard acetonitrile–water solution of glucose and fructose.

To determine the minerals, a wet oxidation procedure was applied. Five g of sample from olive fruit flesh homogenate were placed in a 250 ml preweighed Erlenmayer flask. Concentrated nitric acid (30 ml) was added to each flask; the flask was covered and contents allowed to digest for 24 h. The flask was then placed on a Thermolyne Type hot plate at 130°C to concentrate until the residue was 15 ± 1 g, cooled and transferred into a 100 ml volumetric flask and completed to final volume with distilled deionized water. The blank sample was prepared for corrections applying throughout the entire digestion steps. Standard solutions of each mineral were prepared for the calibration curves under the same conditions. Inductively coupled plasma–mass spectrometry (ICP–MS) (Hewlett Packard 4500) and inductively coupled plasma–flame emission spectrometry (ICP–FES) (Shimadzu AA6701 equipped with Shimadzu HVG-1 hydride vapour generator) were used to determine each element (Al, Cr, Mn, Co, Cd, Ba, Fe, Ca, Mg, Na, K, Cu, Zn and As) in the olive flesh samples. The instrument settings and other experimental conditions were in accordance with the manufacturer's specifications (Anon, 1989a, 1989b). The 'paired *t*-test' was used for statistical treatment of data.

All glassware was washed with detergent, soaked 24 h in 15% (v/v) nitric acid, rinsed with deionised distilled water and dried before use for mineral analysis. All the reagents used were of analytical grade.

3. Results and discussion

Table 1 shows that the number of fruits per kg of Domat variety decreased from September to November and did not change until February. In the variety Memecik, there was an increase varying between 222 and 258 fruit per kg. Flesh to stone weight ratios increased continuously in both olive varieties. Higher ratios were found in Memecik than Domat.

Due to climatic condition, a fluctuation can be observed in moisture content. The moisture content of flesh varied from 52.3 to 69.9% for Domat. There was a rapid increase in October, then it decreased to 55.8%. In Memecik the moisture value continued to increase until November and then a drop occurred (49.4%). Similar behaviour of olive fruit cultivars, moisture content has been reported by Guillen, Fernandez and Hevedia (1993).

The oil accumulation behaviour observed in the flesh of two examined olive varieties is illustrated in Fig. 1.

Table 1
Some physical characteristics of the olive fruit varieties at various stages of ripening[a]

Sampling time	Moisture		Number of fruits per kg		Flesh to stone ratio (*F*/*S*)	
	Domat	Memecik	Domat	Memecik	Domat	Memecik
September	53.2 ± 0.31	48.9 ± 0.46	418	222	2.0 ± 0.14	3.6 ± 0.28
October	66.9 ± 0.23	49.0 ± 0.12	256	258	3.7 ± 0.14	4.8 ± 0.00
November	61.2 ± 0.27	54.5 ± 0.10	250	240	3.8 ± 0.14	4.6 ± 0.14
December	60.9 ± 1.08	49.4 ± 1.41	242	258	3.9 ± 0.14	4.1 ± 0.00
January	55.8 ± 1.80	–	240	–	3.9 ± 0.01	–
February	54.5 ± 0.06	–	240	–	4.1 ± 0.14	–

[a] Average value of three determinations ± S.D.

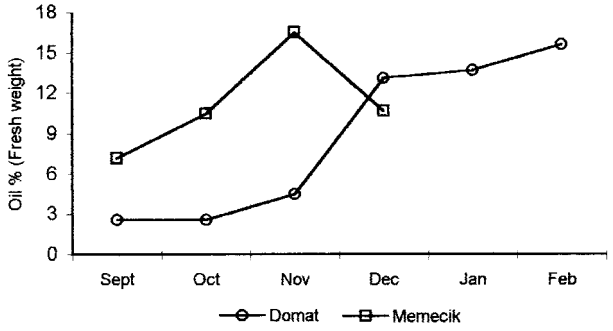

Fig. 1. Oil accumulation in the flesh of olive fruits.

The amount of oil increased in both varieties during the entire sampling period. Only a rapid decrease was observed in the amount of oil for Memecik in December. Fatty acid composition of the oil may differ, depending on the variety of olive and degree of fruit ripeness. As can be seen, numerous fatty acids were present in the examined olive varieties (Tables 2 and 3). Palmitic, stearic, oleic and linoleic were measured as major fatty acids. Palmitoleic, linolenic + eicosenoic acids were also determined in small amounts at all intervals. Myristic, arachidic, behenic and lignoseric acids were present at less than 0.5% in both olive cultivars. The initial linoleic acid contents in Domat and Memecik were 7.4 and 7.71% in September, respectively. Those values increased continuously and reached 16.7% in February and 15.6% in November, respectively. A small reduction was shown in the level of palmitic acid in both cultivars at the end of the sampling period, as compared with its initial value. There was an increase in the percentage of stearic acid of Memecik from September to November and then a slight decrease was observed in December. A regular variation was not found for other fatty acids. Palmitic and oleic acid percentages decreased during the olive ripening, at the same time as quantity of linoleic acid increased (Maestro-Duran,

Table 3
Fatty acid composition of the flesh oil of Memecik variety (%)

Fatty acid	September	October	November	December
Myristic (14:0)	0.006	0.008	0.011	0.017
Palmitic (16:0)	15.0	14.7	14.8	13.9
Palmitoleic (16:1)	1.60	1.35	0.94	1.01
Margaric (17:0)	0.14	0.07	0.04	0.04
Stearic (18:0)	2.23	2.47	2.73	2.53
Oleic (18:1)	71.5	68.0	63.7	67.0
Linoleic (18:2)	7.7	11.6	15.6	13.7
Arachidic (20:0)	0.40	0.41	0.42	0.37
Linolenic + cis-11-eicosenic (18:3) + (20:1)	0.94	0.95	1.11	0.92
Behenic (22:0)	0.13	0.16	0.12	0.08
Lignoseric (24:0)	0.13	0.12	0.16	0.25

Table 2
Fatty acid composition of the flesh oil of Domat variety (%)

Fatty acid	September	October	November	December	January	February
Myristic (14:0)	0.01	0.01	0.01	0.01	0.004	0.003
Palmitic (16:0)	14.6	15.0	14.1	15.0	13.5	13.7
Palmitoleic (16:1)	1.82	1.26	1.19	1.12	1.00	0.96
Margaric (17:0)	0.42	0.30	0.24	0.21	0.16	0.20
Stearic (18:0)	4.36	3.98	3.83	3.32	3.42	3.25
Oleic (18:1)	68.2	63.5	65.3	63.5	64.3	62.8
Linoleic (18:2)	7.4	14.3	13.6	14.8	15.6	16.7
Arachidic (20:0)	0.38	0.36	0.33	0.28	0.24	0.25
Linolenic + cis-11-eicosenic (18:3) + (20:1)	0.49	0.51	0.53	0.55	0.58	0.56
Behenic (22:0)	0.43	0.18	0.11	0.17	0.19	0.19
Lignoseric (24:0)	0.91	0.14	0.27	0.25	0.66	0.84

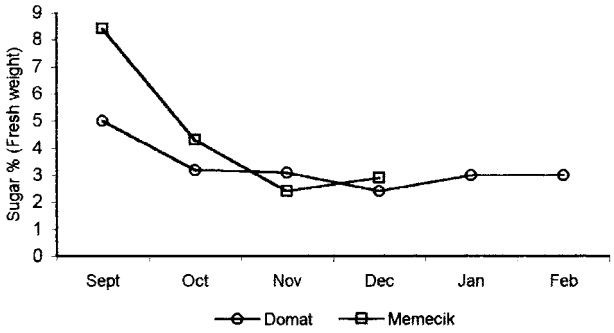

Fig. 2. Total sugar content of olive fruits.

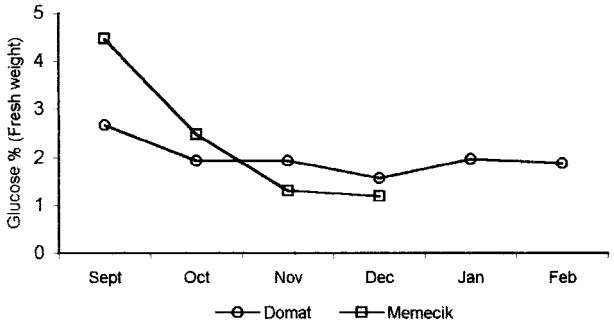

Fig. 3. Amount of glucose in olive fruits during ripening.

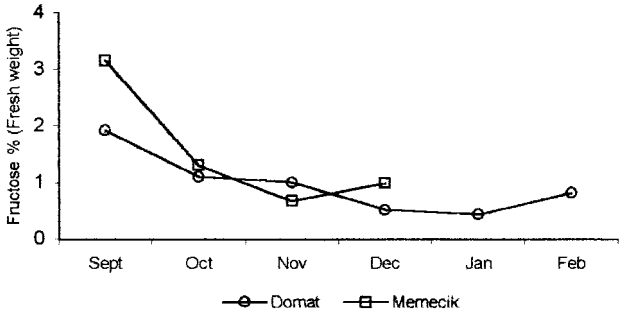

Fig. 4. Amount of fructose in olive fruits during ripening.

1990). No reported literature was found concerned with variations other than major fatty acid percentages of olive fruit during ripening. For this reason, our results cannot be compared with existing literature. In general, oleic and palmitic acid contents of the olive flesh decrease while linoleic acid increases as olive ripens.

The amounts of total sugar and individual sugars were determined in the examined cultivars. Total sugar contents in the cultivars of Domat and Memecik at full maturation were 3.0 and 2.9%, respectively (Fig. 2). Initial sugar content of Domat variety in September was lower than Memecik. In both cultivars, sugar content decreased continuously until November and then increased up to the value of 3.0%. These results are in agreement with those reported by Wodner et al. (1987) and Patumi et al. (1990). The concentration of individual sugars, glucose and fructose, at the various stages of development for both cultivars, are shown in Figs. 3 and 4. The level of glucose in Domat decreased to a minimum level of 1.57% and then increased. Fructose level also decreased during the sampling period to 1.19%. The lowest value for Memecik was 0.66% in November, after which fructose content increased and reached 0.98%. Glucose and fructose were predominant sugars in olive fruits. In general, fructose content was found to be smaller than that of glucose in all stages of maturation. In the present study, a relationship was found between the oil accumulation and sugar content in the examined olive cultivars. A positive correlation was found to be statistically significant ($P \leqslant 0.05$). From the results, it can be said that the carbohydrates may be precursors for fatty acid biosynthesis in the olive fruits. However, other factors such as malate and citrate of olive fruits should also be considered for lipid biosynthesis.

The results from analysis of minerals in olive flesh show that overall composition of minerals varied markedly among the cultivars. The amounts of potassium,

Table 4
Mineral content of Domat variety olives

Minerals	September	October	November	December	January	February
Na (ppm)	11.1	11.8	17.2	19.2	20.0	32.8
K (ppm)	16 666	13 951	14 516	14 397	15 438	15 187
Ca (ppm)	56	40	25	23	24	28
Mg (ppm)	160	135	109	114	136	142
Cu (ppm)	3.4	2.1	2.4	2.2	2.4	2.1
Zn (ppm)	5.2	3.1	3.3	3.6	3.7	3.7
Mn (ppm)	1.91	1.31	1.26	1.21	1.24	1.34
As (ppb)	109	77	76	73	70	68
Ba (ppb)	319	268	225	90	< 50	< 50
Cr (ppb)	219	129	106	92	87	75
Co (ppb)	15	9	8	9	9	10
Cd (ppb)	35	18	17	12	6	< 5
Fe (ppm)	16.5	13.6	10.9	10.2	10.0	9.5
Al (ppm)	3.4	2.6	1.5	2.5	2.6	5.7

Table 5
Mineral content of Memecik variety olives

Minerals	September	October	November	December
Na (ppm)	35.4	32.9	39.6	43.0
K (ppm)	14 318	15 367	15 374	15 820
Ca (ppm)	604	650	413	489
Mg (ppm)	271	372	269	344
Cu (ppm)	8.4	3.5	2.8	2.3
Zn (ppm)	10.0	5.8	4.5	6.7
Mn (ppm)	1.21	0.87	0.78	0.67
As (ppb)	128	128	110	63
Ba (ppb)	431	465	461	513
Cr (ppb)	74	58	46	53
Fe (ppm)	8.1	5.8	5.6	11.4
Al (ppm)	0.9	2.6	3.2	7.3

sodium, magnesium, calcium and iron were determined to be highest in both cultivars. From an examination of Tables 4 and 5, it is obvious that potassium is the most abundant element in the fruit, followed by Mg, Ca, Na and Fe in Domat. Potassium in Memecik is the same as in Domat cultivar. The other elements, in descending order by quantity, were Ca, Mg and Fe. The level of sodium in both cultivars increased continuously from September to January. It was also found that the content of potassium of olive fruit flesh in Memecik increased during the sampling period. The variations of elemental concentration in the olive flesh during ripening were not uniform. While some elements showed a steady increase at all intervals, others showed a decrease. These variations can be originating from olive varieties, distribution of elements in the soil, as well as environmental and weather conditions during the sampling period. The present data are important in providing information about the compounds of olive fruit appearing during maturation and as precursors for fatty acid synthesis, since few facts in the literature are available to explain these phenomena in olive fruits.

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