# INFLUENCE OF OLIVE MATURITY ON SOME PHYSICO-CHEMICAL PROPERTIES AND FATTY ACID COMPOSITION OF MONOVARIETAL OLIVE OIL EXTRACTED FROM HALHALI CULTIVAR

DILSAT BOZDOGAN KONUSKAN

*Mustafa Kemal University, Faculty of Agriculture, Food Engineering Department, dilsat@mku.edu.tr*

This study was carried out to determine influence of olive maturity on some physicochemical properties and fatty acid compositions of olive oils extracted from the Halhalı cultivar which harvested from Hatay in the Eastern Mediterranean region of Turkey. For this purpose, olive oils were obtained by mechanical method from olives collected from Halhalı cultivar in 3 different olive maturity of the 2021 production season. Ripening index and oil yield analysis of the olives and free fatty acids, peroxide value, fatty acid compositions were carried out in Halhalı olive oil. Free fatty acids and peroxide values of olive oils were found in the range of 0.39-0.73 (%oleic acid) and 5.14-9.43 meq $O_2$/kg respectively. The amount of free fatty acids increased with maturity. It was determined that the oleic acid in the range of 67.59%-70.26%, palmitic acid in the range of 13.56-15.82%, linoleic acid in the range of 9.52-13.65%, stearic acid in the range of 3.34-4.13%, palmitoleic acid 0.96%-1.29%, linolenic acid 0.86-0.98% and arachidic acid 0.42-0.53. It was determined that decrease in oleic acid and palmitic acid contents and an increase in linoleic acid content with maturity. It has been determined that Halhalı monovarietal olive oil is within the limits specified in the Turkish Food Codex on Olive Oil and Pirina Olive Oil in terms of the examined properties.

Keywords: Olive oil, ripening, fatty acids

## INTRODUCTION

Virgin olive oil (VOO) has nutritional and functional characteristics that make it unique among other vegetable oils (Baieno *et al*., 2015). Virgin olive oil is mainly composed of two different fractions: the saponifiable fraction and the unsaponifiable fraction. The saponifiable fraction represents nearly 98% of the total composition, including the fatty acids and triacylglycerols (Visioli and Galli 2002; Ranalli *et al*., 2004). The main components in the unsaponifiable fraction are sterols, alcohols, vitamin E, hydrocarbons, carotenoids, volatile compounds and phenolic compounds, representing only 2% of the total (Antonini *et al*., 2015). Olive oil has a source of monounsaturated fatty acids, especially oleic acid (60–80%), which is less susceptible to oxidation, having an important role in terms of contributing to the high stability and long shelf life of olive oil (Anastasopoulos *et al*., 2012). Several factors such as variety, ripening degree, climate and geographic conditions, extraction methods, and preservation conditions significantly influence the chemical composition and quality of VOO (Salvador *et al*., 2013; Tura *et al*., 2007; Konuskan and Mungan, 2016). Quality properties and fatty acid composition are significantly affected by olive fruit maturity. Maximum oil content is reported to occur between 60th to 75th days after the start of the ripening process (Aloiwaiesh *et al*., 2018). The objective of this study was to evaluate the influence of olive maturity and on Halhalı monovarietal olive oil quality and fatty acid compositions.

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## EXPERIMENTAL

### Olive Sampling

This study was conducted during the crop season of 2021. Halhalı olive variety obtained from three single trees of a given variety were collected from in Hatay (Fig. 1). Only undamaged fruits which were considered healthy were hand-picked from each younger tree. Three harvesting dates (from October to December with 20 days intervals) corresponding to three different ripening stages (green–spotted–ripe) was selected. At the end of each harvest, the samples were labelled and immediately transported to the laboratory. They were extracted to olive oil within 24 h.

Figure 1. Halhalı variety tree in Hatay

A representative 3 kg olive sample was extracted to obtain the oil by a laboratory scale mechanical mill (Hakkı Usta, Turkey) with a crusher, a vertical malaxer and a two-phase centrifuge. Malaxation and centrifuge processes was performed at 28 °C for 45 min and at 3000 rpm, respectively. The oil was separated by decanting and put into dark glass bottles. Oil samples were stored in darkness at 4 °C until chemical analysis which were triplicated.

### Ripening Index

The ripening index (RI) was determined from one hundred olive fruits randomly drawn from each olive variety. This parameter, which is based on evaluating the color of both skin and pulp of olives, was determined according to International Olive Council (IOOC 2001).

### Oil Content (%)

Oil content was determined according to the method described in American Oil Chemists' Society (AOCS) Official Methods Am 2–93 (AOCS, 2003) by Soxhlet extraction method using n-hexane at 80°C for 6 h.

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**Olive Oil Extraction**

Three kg of olive samples selected from Halhalı variety were eliminated from unhealthy and decay fruits. Olive oil extraction (Figure 2) was performed using a laboratory scale mechanical extractor. It is equipped with a crusher, a malaxer and a decanter. In fact, olives were crushed and then slowly kneaded for 40 min at 28°C. Next, the obtained paste was centrifuged at 3000 rpm for 2 min with the decanter. The oil was put into dark glass bottles under a nitrogen atmosphere of 100 ml without headspace. The oil obtained was kept at 4°C in the dark area until analyses which were duplicated.

Figure 2. Olive oil extraction

**Fatty Acid Composition**

Fatty acid composition of Halhalı olive oils was determined according to the analytical method described in by the International Olive Oil Council, COI/T.20/Doc.No.24 (IOOC, 2004). Methyl-esters were prepared by vigorous shaking of a solution of oil in n-heptane (0.1 g in 2 mL) with 0.2 ml of 2 N methanolic potassium hydroxide and analyzed by Agilent gas chromatography system (Agilent 6850, USA) equipped with a hydrogen flame ionization detector (FID) and a capillary column DB-23 of 60 m length × 0.25 mm i.d. and 0.25 µm of film thickness. The carrier gas was helium at 1.0 ml/min ratio. Injector, oven and detector temperatures were 250, 230 and 280 °C, respectively. The injection volume was 1 µL. The results were expressed as a relative area percentage of total fatty acid methyl esters. Fatty acids were determined by comparing their retention times with those of reference compounds.

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## RESULTS AND DISCUSSION

### Ripening Index

Table 1 reports the ripening index (RI) and oil content of Halhalı variety. The RI values was determined in the range of 0.75 (green) – 4.56 (ripe). RI values were increased with the ripening degree. RI analysis are very important for determining the harvesting time during the fruit ripening process (Yang *et al*., 2021).

### Oil Content (%)

As can be seen in Table 1, the oil content of Halhalı olives varied between 27.03 (green) and 34.19% (ripe). The oil content of Halhalı olives increased remarkably from green to ripe maturation stage. In similar studies, this value was found to be 16.19-33.93% by Yorulmaz and Bozdogan Konuskan (2017) and 24.69-41.70% by Emmanouilidou *et al*. (2021).

Table 1. Physicochemical properties of Halhalı monovarietal olive oil

| Physicochemical properties | Green | Spotted | Ripe |
|---|---|---|---|
| Ripening Index | 0.75±0.06 | 2.58±0.03 | 4.56±0.06 |
| Oil content (%) | 27.03±1.24 | 30.16±1.36 | 34.19±2.17 |
| Free fatty acids (%Oleic) | 0.39±0.07 | 0.58±0.16 | 0.73±0.22 |
| Peroxide value (meq$O_2$/kg) | 5.14±0.61 | 8.02±0.52 | 9.43±0.18 |

### Free Faty Acids (% Oleic)

As can be seen in Table 1, free fatty acids of olive oils ranged between %0.39 (green)–%0.73 (ripe). Moreover, the free fatty acid content of oils was below the limit of 0.8% established by Turkish Food Codex and Regulation EC/ 1989/2003 (European Union Commission, 2003; Turkish Food Codex, 2014). The free fatty acids of olive oils increased with the olive maturity. Moreover, the free fatty acid percentages of our olive oil samples were higher than those of the values reported by Emmanouilidou *et al*. (2021). De Mendoza *et al*. (2013) reported that the related the highest acidity value of olive oils during olive maturation to the progressive action of the lipolytic activity.

### Peroxide Value

Peroxide values varied between 5.14 (green) and 9.43 (ripe) meq$O_2$/kg oil and the peroxide values of olive oils were below 20 meq/$O_2$ kg which is accepted as the legal limit for extra virgin olive oils by Turkish Food Codex and Regulation EC/1989/2003 (European Union Commission, 2003; Turkish Food Codex, 2014). The peroxide values increased with significantly throughout maturation. These results were in accordance with those of Polari *et al*. (2020).

### Fatty Acid Composition

The fatty acids composition of Halhalı olive oils are shown in Table 2. As shown in Table 2. major fatty acids that were found in olive oils were palmitic (C16:0), oleic (C18:1), linoleic (C18:2) acid. The minor fatty acids were palmitoleic (C16:1), stearic (C18:0), linolenic (C18:3), arachidic (C20:0), gadoleic (C20:1), behenic (C22:0) acids. Olive oil characterized by a high level of oleic acid. The main fatty acid in Halhalı olive oil

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was oleic acid, ranging from 67.59 % (ripe) to 70.26 % (green) and it was determined that there was a decrease in oleic acid content with olive maturation. Our results showed similarity with those of the results obtained by Navajas Porras *et al*. (2020). Palmitic acid, which was the second most abundant fatty acid, ranged from 13.56 (ripe) to 15.82% (green). It was determined that the palmitic acid values of the Halhalı olive oils decreased with the olive ripening. Linoleic acid content ranged between 9.52 % (green) and 13.65 % (ripe). Linoleic acid content increased during maturation, confirming the results reported by Konuskan and Mungan (2016). Palmitoleic, linolenic, arachidic, gadoleic and behenic acids were between 0.96–1.29, 0.86–0.98, 0.42–0.53, 0.19–0.27 and 0.09-0.14 %, respectively. The fatty acid compositions of oils from Halhalı monovarietal olive oils were within the legal limits established by Turkish Food Codex and Regulation EC/ 1989/2003 (European Union Commission, 2003; Turkish Food Codex, 2014). The fatty acid composition of olive oil is significantly influenced by the cultivar, olive maturation, growing region (Salvador *et al*., 2003; Yorulmaz and Bozdogan Konuskan, 2017; Alowaiesh, 2018).

Table 2. Fatty acid compositions (%) of Halhalı monovarietal olive oil

| Fatty acids (%) | Green | Spotted | Ripe |
|---|---|---|---|
| Palmitic acid (16:0) | 15.82±0.46 | 14.09±0.88 | 13.56±0.79 |
| Palmitoleic acid (16:1) | 1.29±0.27 | 1.02±0.44 | 0.96±0.08 |
| Stearic acid (18:0) | 3.34±0.12 | 3.92±0.67 | 4.13±0.53 |
| Oleic acid (18:1) | 70.26±1.25 | 68.81±0.94 | 67.59±0.88 |
| Linoleic acid (18:2) | 9.52±0.32 | 11.23±0.24 | 13.65±0.69 |
| Linolenic acid (18:3) | 0.86±0.03 | 0.91±0.22 | 0.98±0.02 |
| Arachidic (C20:0) | 0.42±0.01 | 0.48±0.03 | 0.53±0.08 |
| Gadoleic (C20:1) | 0.27±0.07 | 0.21±0.06 | 0.19±0.05 |
| Behenic (C22:0) | 0.14±0.01 | 0.09±0.04 | 0.11±0.03 |

## CONCLUSIONS

As a result, it has been determined that the changes in physicochemical properties and fatty acid compositions of monovarietal of olive oil extracted from Halhalı cultivar depending on different olive maturation. As maturation progressed, a series of changes occurred in olive oil samples and with an influence especially on some parameters such as oil content, free fatty acids and fatty acid composition. The results of this study showed that olive maturation had an important role on the physicochemical properties and fatty acid compositions of olive oils.

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