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Summary

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Influences of filtration and storage time on the quality of Tavşan Yüreği extra virgin olive oil (EVOO)

Einflüsse der Filtrations- und Lagerzeit auf die Qualität von Tavşan Yüreği Natives Olivenöl extra (EVOO)

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In this study, effects of filtration and storage time on the chemical composition and sensory profile of the virgin olive oil extracted from a local olive cultivar named as Tavşan Yüreği grown in Antalya province of Turkey were investigated. The study was carried out during the crop year 2014/2015. "Tavşan Yüreği" olives were collected at early harvest stage and processed into virgin olive oil by using a state-of-the-art mobile olive mill (MOM) equipped with a knife crusher, malaxer and 2-phase decanter. Changes in the free fatty acid composition and peroxide values, UV absorption values, total phenol content, phenolic and tocopherol profiles of filtered (F) and unfiltered (UF) samples were monitored monthly during 12 months of storage. Results showed that both F and UF olive oil samples were classified as EVOO according to the International Olive Council (IOC) standards. Although UF olive oil had higher total phenols content than the filtered sample at the beginning of storage, total phenol contents were comparable in both F and UF olive oils after a year storing. Phenolic and tocopherol contents decreased in all samples with storage time. The results obtained in this study showed that Tavşan Yüreği olive oils has a unique chemical composition as well as good oxidative stability. On the other hand, filtration caused a decrease in the total polyphenol content. Although filtration process had no significant effect on oxidation stability parameters such as free fatty acidity, and peroxide values, but filtration resulted in lighter colored oils. Generally, both filtration process and storage time caused to significant decreases in the amount of tocopherol and total polyphenolic contents of EVOO samples ($P < 0.01$).

Abbreviation: F= Filtered monocultivar olive oil was extracted from Tavşan Yüreği UF= Unfiltered monocultivar olive oil were extracted from Tavşan Yüreği, EVOO= Extra Virgin Olive Oil.

Keywords: Olive oil, Tavşan Yüreği, Stability, Storage, Filtration

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Introduction

Virgin olive oil (VOO) is defined that it is the oil obtained from fruits of the olive tree solely by mechanical or other physical methods under conditions which do not alter the oil in any way (IOC method COI/T.15/NC No 3/ Rev. 7 May 2013). This means virgin olive oil is produced by the exclusion of extraction with solvents or re-esterification processes and of any mixture with other cheap oils. So, virgin olive oil is the juice of the olive fruit and it is ready to be consumed almost entirely in its natural state. Indeed, olive oil extraction is a process of separating the oil from the other fruit contents (vegetation tissue, water, and solid material) with the following steps: washing, crushing, malaxation (mixing) and separation of the oil. The purpose of washing is to remove any foreign material that could damage machinery or contaminate the oil. Crushing the olives is to produce a paste with easily extracted oil droplets. Malaxation prepares the paste for separation of the oil from the pomace and optimizes oil yield through the formation of larger oil droplets and a reduction of the oil-water emulsion. The next step is extracting the oil from the paste and fruit water (water of vegetation). The oil can be extracted by pressing, centrifugation, percolation, or through combinations of different methods. After the extraction, olive oil generally needs cleaning operations using a vertical disc stack centrifuge separator and/or a filter for removing suspended solids, removing humidity and making the oil transparent and clear appearance. Indeed filtration is one of the important steps that cause qualitative and quantitative changes, especially on minor components affecting of VOO quality in terms of health benefits, shelf life, and sensory properties.

Storage of olive oil under nitrogen pressure in a dark place at room temperature (25–30°C or lower) increases shelf life (Boskou, 2006). A decrease in chlorophyll, carotenoid contents were also reported (Romero et al., 2004). α -tocopherol loss reached up to 79% in four months, whereas <45% of the phenols were lost under diffused light during storage (Okogeri and Tasioula-Margari, 2002). A positive correlation was found between the age of the oils and the tyrosol to total phenols ratio (Cinquanta et al., 1997). EVOO with high antioxidant contents was still “good quality” after 240 days of storage at 40 °C (Lavelli et al., 2006). Although an important loss of total phenol content was seen in commercial Arbequina virgin olive oil after 12 months of storage, no changes in some phenolics (tyrosol and hydroxytyrosol) and aromatic hydrocarbon contents of freeze samples were reported up to 12 months (Mulinacci et al., 2013). Psomiadou et al. (2000) suggested good handling is quite important to keep high α -tocopherol levels of Greek VOO under domestic conditions for two years.

Some studies showed that filtration has positive effects such as increasing oil stability by reducing moisture and free fatty acidity, decreasing the rate of secoiridoid hydrolysis that can affect shelf life over time, eliminating undesired volatile compounds that affect the aroma of the oil, reducing rancidity of the oil, removing muddy sediment defect, contributing to clear appearance, lowering the amount of pigments thus reducing to susceptibility to photooxidation (Lozano-Sanchez et al., 2010; Breschi et al., 2019). On the other hand, filtration has some negative effects such as: decreasing oil stability due to removal of suspended solids and exposure to oxygen during filtration, decreasing water-soluble phenolic content and antioxidants that help prevent oxidation, eliminating desired volatile compounds

that affect the aroma of the oil, decreasing positive attributes (fruitiness, bitterness, pungency), contributing to lighter appearance and lower intensity of the green color, decreasing pigment concentrations and limiting the ability to capture free radicals in dark, reducing shelf life due to decrease in phenolic content (Ngai and Wang, 2015). In contrast, some studies showed increases in the contents of some phenolics (oleuropein and ligstroside derivatives) in the filtered oils after vertical centrifuge treatment (Bakhouche et al., 2014). Sinesio et al. (2015) reported that unfiltered VOO with high polyphenol content showed a significant modification of the sensory characteristics after four months and decantation systems (two-phase or three-phase) had no effect on the sensory properties. Brkić Bubola et al. (2017) reported that filtration provided a more stable sensory profile as compared to veiled olive oil. These results showed that the literature lacks consensus regarding the effects of filtration on the oxidative stability, chemical and sensory properties of VOO during storing. Thus, it is necessary to examine the effect of filtration on the properties of VOO during storage in detail.

Tavşan Yüreği olive is originated and located mainly Döşemealtı and Kepez villages of Antalya province, located in the Western Mediterranean Region (Figure. 1). In this study, we aimed to perform the best processing method for olive oil production from Tavşan Yüreği. For this purpose, a mobile olive oil processing unit was used for cold press olive oil production. Quality parameters were determined pre and post-filtration, and the changes were also monitored during 12 months' storage.

Methods and material

Production of Extra Virgin Olive Oil (EVOO)

A “Mobile Olive Oil Processing Unit” (MOOPU) with state-of-the art Olemio equipments was used in order to produce premium quality EVOO. The MOOPU is a unique olive extraction unit (500 kg/h capacity) equipped with a knife crusher and a two-phase horizontal decanter (Oliomio D500, Italy). The mobile unit is an articulated lorry with a special semi-trailer measuring 2438 x 12 192 x 2896 mm which is divided into three separate sections. The first section is olive accepting unit including; bunker, leaf removers, washer and crusher units of the system. The second section is processing unit including malaxer, decanter, filter and bag-in-box filling machine. The third section is support unit placed a power plant and a water supply tank. Processing unit is a hygienic area so protected for temperature changes, dust and odor. This hygienic area was equipped by an air conditioner, isolation and filter ventilation systems. Tavşan Yüreği olive cultivar grown locally in Antalya was harvested by hand picking (about one metric ton) in the early harvest period during 2014–2015 season and processed in the MOOPU in a few hours. Olive paste was prepared to crush by a hammer mill and the paste was mixed in the malaxer at 27°C for 15 min. After decantation (about 100 liters) EVOO was packaged before (Unfiltered) and after filtration (Filtered). A filter press (Oliomio Jolly 40, Italy) with the paper (Gruppo Cardenons E2, paper weight: 350 g/m², thickness: 0,81 mm, apparent density: 0.43 g/cm³, water absorption: 8 g/dm²) was used for filtration. Olive oil samples were filled in 250 ml amber glass bottles (headspace: 4 cm) by nitrogen gas. Total 24 bottles (12 of them for chemical and 12 of them for sensorial analyses) were stored at room temperature (18–24 °C) up to 12 months.

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Analytical Methods

Free fatty acid content and peroxide value were performed according to the EEC 2568/91 and AOCS Cd 8-53 methods, respectively. Moisture content was determined according to the ISO 662. Color values (L , a , b values) were determined by spectrophotometer (Minolta, CM-3600d, Japan). The maximum for L is 100, which would be a perfect reflecting diffuser. The minimum measuring for L would be zero, which would be black. UV absorbance was performed according to the IOC method COI/T.20/Doc. No 19/Rev. 3. UV absorbance was measured by using UV Spectrophotometer (Agilent 8453, USA).

Total Phenolic Content

The polar fraction was extracted and used for total phenolic and phenolic composition analyses. 2.5 gram of olive oil sample was weighed into a falcon tube. Hexane (6 ml) was added and shaken for 1 min. This solution was filtered through solid phase extraction (SPE) cartridge (Superclean LC-Diol, USA) and collected in a glass tube. Then hexane (6 ml) and 4 ml hexane: ethyl acetate (85:15, v/v) were passed through the SPE cartridge, respectively. The cartridge was washed with of methanol: deionized water solution (1:1 v/v) and the phenolic extract was evaporated (UniEquip Univapo 100 ECH, Canada). After addition of 2 ml methanol: deionized water solution (1:1 v/v) the tubes vortexed for 30 second. For determination of total phenols, Folin & Ciocalteu method was used and the results were expressed in terms of gallic acid equivalent (mg gallic acid/kg oil) (Romani et al., 2007; Inarejos-Garcia et al., 2009). Ultra high-performance liquid chromatography (UHPLC, Thermo Scientific Dionex Ultimate 3000, USA) and C18 column (4.6 mm inner diameter x 250 mm length and 5 mm particle diameter; Thermo scientific acclaim 120) was used for determination of phenolic profile. Prepared phenolic extract (1 ml) was passed through 0.45 μ m microfilter (Merck, PVDF, Millipore Millex-HV, Germany) and poured into an amber vial. Column temperature was

fixed at 30°C and acetic acid: deionized water (1:1) (A), methanol (B), acetonitril (C) were used in a gradient flow program as mobile phase. In the gradient program eluents were 2.5 % B, 2.5 % C, and 95% A solution up to 60 min. Flow rate was 1ml/min and diode array detector (DAD) detector was set in 280 nm, 320 nm and 335 nm. Apigenin, caffeic acid, gallic acid, luteolin, m-cumaric acid, p-coumaric acid, oleuropein, syringic acid, trans-ferulic acid, vanillic acid, vanillin, tyrosol, 3-hydroxy tyrosol, 3,4-dihydroxy benzoic acid, 4-hydroxy benzoic acid, 4-hydroxy phenyl acetic acid were used as standards.

Tocopherol Composition

EVOO sample (2 g) was weighed into a 25 ml volumetric flask (AOCS Official Method Ce 8-89, 1997. A quantity of hexane was added and shaken to dissolve the sample. Flask was made up to volume with the same solvent. Solution was passed from syringe filter (0.45 μ m) (PVDF, Millipore Millex-HV) into the HPLC vial. The samples (20 μ L) injected to HPLC (UHPLC: Ultra High Performance Liquid Chromatography (Dionex Ultimate 3000). LiChrosorb SI 60-5 column (4.6 mm I.D x 250 mm length and 5 μ m particle size) was used for analysis. Column temperature was fixed at 30°C during the process. Flow rate of analysis was 1 mL/min. For mobile phase, isopropanol: hexane (0.5 :99.5, v/v) isocratic mix was used and chromatograms were collected at 292 nm wavelength. Analysis time was 30 min and the injection volume was 100 μ L. Amounts of α , β , γ and Δ tocopherols were determined by using tocopherol standards.

Sensory Analysis

Sensory analysis of olive oil samples was performed by the Ayvalık Olive Oil Tasting Laboratory accredited by International Olive Council and TURKAK (Turkish Accreditation Agency) according to the method for the organoleptic assessment of virgin olive oil (COI/T.20/Doc. No. 15/Rev. 8, November 2015). Eight trained tas-

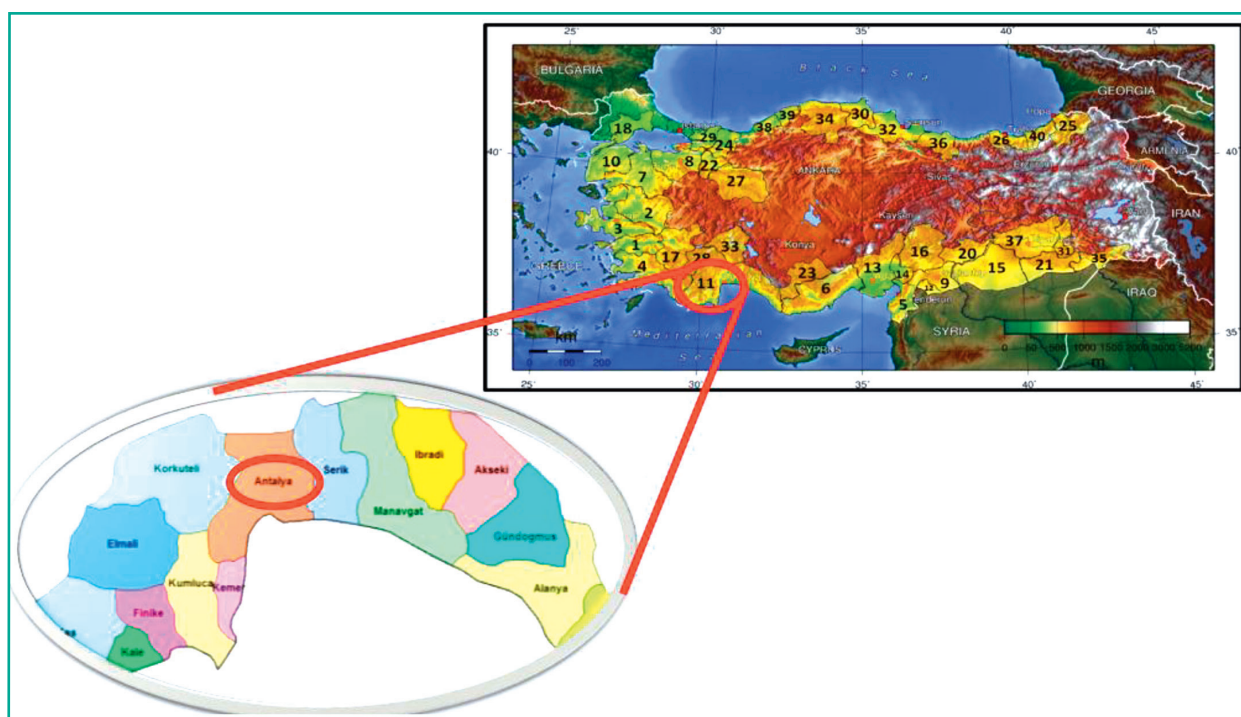


FIGURE 1: Map of Antalya province.

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TABLE 1: Oxidative stability parameters of extra virgin olive oils extracted from Tavşan Yüreği variety during 12 months storage.

Storage period (month)	Free Fatty Acid (%)		Peroxide Value (meq O ₂ /Kg Oil)		K ₂₃₂		K ₂₇₀	
	F	UF	F	UF	F	UF	F	UF
0	0.1±0.00 ^{Ab}	0.1±0.00 ^{Ab}	8.99±0.006 ^{Bi}	11.94±0.016 ^{Ae}	1.8±0.00 ^{Bd}	2.0±0.00 ^{Ab}	0.11±0.00 ^{Bh}	0.15±0.00 ^{Af}
1	0.1±0.00 ^{Ab}	0.1±0.00 ^{Ab}	8.99±0.000 ^{Bi}	11.94±0.000 ^{Ae}	1.7±0.00 ^{Ae}	1.7±0.00 ^{Af}	0.15±0.00 ^{Bf}	0.17±0.00 ^{Ad}
2	0.1±0.00 ^{Ab}	0.1±0.00 ^{Ab}	11.80±0.012 ^{Bh}	11.95±0.004 ^{Ae}	1.5±0.00 ^{Bf}	1.6±0.00 ^{Ag}	0.11±0.00 ^{Bh}	0.16±0.00 ^{Ae}
3	0.1±0.00 ^{Ab}	0.1±0.00 ^{Ab}	11.96±0.001 ^{Bg}	11.98±0.001 ^{Ad}	1.4±0.00 ^{Bg}	1.5±0.00 ^{Ah}	0.09±0.00 ^{Bi}	0.18±0.00 ^{Ac}
4	0.1±0.00 ^{Ab}	0.1±0.00 ^{Ab}	14.95±0.003 ^{Bf}	14.99±0.000 ^{Ac}	1.4±0.00 ^{Ag}	1.4±0.00 ^{Ai}	0.17±0.00 ^{Ad}	0.14±0.00 ^{Bg}
5	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	14.97±0.013 ^{Be}	14.99±0.000 ^{Ac}	1.9±0.00 ^{Bb}	2.0±0.00 ^{Ab}	0.18±0.00 ^{Bc}	0.20±0.00 ^{Ab}
6	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	14.99±0.001 ^{Ade}	14.99±0.000 ^{Ac}	1.9±0.00 ^{Bb}	2.0±0.00 ^{Ab}	0.19±0.00 ^{Bb}	0.20±0.00 ^{Ab}
7	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	14.99±0.009 ^{Ade}	14.99±0.003 ^{Ac}	1.8±0.00 ^{Bd}	1.9±0.00 ^{Ad}	0.16±0.00 ^{Ae}	0.15±0.00 ^{Bf}
8	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	15.01±0.010 ^{Ad}	15.01±0.019 ^{Ac}	2.2±0.00 ^{Ba}	2.3±0.00 ^{Aa}	0.20±0.00 ^{Aa}	0.20±0.00 ^{Ab}
9	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	17.97±0.011 ^{Ab}	17.98±0.017 ^{Aa}	1.4±0.00 ^{Bg}	1.6±0.00 ^{Ag}	0.10±0.00 ^{Bi}	0.21±0.00 ^{Aa}
10	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	17.99±0.001 ^{Aa}	17.99±0.002 ^{Aa}	1.8±0.00 ^{Ad}	1.8±0.00 ^{Ae}	0.14±0.00 ^{Ag}	0.14±0.00 ^{Ag}
11	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	17.99±0.009 ^{Ab}	17.99±0.006 ^{Aa}	1.8±0.00 ^{Ad}	1.7±0.00 ^{Bf}	0.11±0.00 ^{Ah}	0.11±0.00 ^{Ah}
12	0.2±0.00 ^{Aa}	0.2±0.00 ^{Aa}	17.78±0.019 ^{Bc}	17.84±0.027 ^{Ab}	1.8±0.00 ^{Bc}	1.9±0.00 ^{Ac}	0.10±0.00 ^{Bi}	0.15±0.00 ^{Af}

*Different superscript letters in the same column indicate significant difference between mean values ($P < 0.01$). **Different superscript uppercase letters in the same row indicate significant difference between mean values ($P < 0.01$).

ting panels were able to assess the oils to determine the levels of positive attributes, such as fruitiness, bitterness and pungency. Negative attributes arising due to poor quality fruit, incorrect processing or storing, such as rancidity, musty and fusty, were determined by sensory panels. Descriptors were evaluated on a 0–10 intensity scale (a number between 0 and 10).

Statistical Analysis

Statistical analysis was performed by SPSS 17 (SPSS Inc. Chicago, IL) statistical software and using One-way ANOVA method. All analyses were performed at least duplicate and differences among all groups were determined by Duncan test.

TABLE 2: Color values (*L*, *a*, *b* values) of filtered and unfiltered of extra virgin olive oils extracted from Tavşan Yüreği variety during 12 months of storage period.

Storage period (month)	<i>L</i> value		<i>a</i> value		<i>b</i> value	
	F	UF	F	UF	F	UF
0	32.88±0.692 ^{Ade}	31.17±0.077 ^{Bef}	0.12±0.007 ^{Bh}	0.43±0.021 ^{Ad}	12.97±1.781 ^{Aa}	4.65±0.021 ^{Bde}
1	34.62±1.421 ^{Abc}	25.69±0.014 ^{Bg}	0.48±0.014 ^{Bg}	0.57±0.067 ^{Ad}	10.55±2.255 ^{Aa}	2.77±0.247 ^{Bf}
2	32.42±0.148 ^{Ae}	30.95±0.530 ^{Bf}	0.72±0.056 ^{Bf}	1.35±0.007 ^{Ac}	13.78±0.240 ^{Aa}	8.37±0.353 ^{Bbc}
3	35.70±0.233 ^{Ab}	32.50±1.675 ^{Bcde}	0.86±0.014 ^{Bef}	1.24±0.134 ^{Ac}	12.70±0.332 ^{Aa}	10.06±0.890 ^{Bbc}
4	33.89±0.374 ^{Acd}	33.80±0.212 ^{Abc}	0.89±0.035 ^{Bdef}	1.43±0.017 ^{Ac}	12.09±3.224 ^{Aa}	9.97±0.381 ^{Bbc}
5	35.63±0.084 ^{Ab}	33.18±0.219 ^{Bcd}	1.08±0.021 ^{Bcd}	1.55±0.141 ^{Abc}	11.90±0.226 ^{Aa}	9.73±2.156 ^{Bbc}
6	35.87±0.190 ^{Ab}	32.05±0.876 ^{Bdef}	-0.06±0.028 ^{Bh}	1.47±0.215 ^{Ac}	12.96±1.385 ^{Aa}	9.05±3.217 ^{Bbc}
7	35.09±0.056 ^{Abc}	33.12±0.657 ^{Bcd}	1.00±0.021 ^{Bcde}	1.57±0.000 ^{Abc}	11.69±0.339 ^{Aa}	9.23±0.169 ^{Bbc}
8	45.06±0.028 ^{Aa}	44.57±0.134 ^{Ba}	2.03±0.010 ^{Ba}	2.73±0.035 ^{Aa}	17.44±0.240 ^{Aa}	16.89±0.367 ^{Ba}
9	33.92±0.692 ^{Acd}	33.14±0.890 ^{Acd}	1.12±0.031 ^{Bc}	1.49±0.003 ^{Ac}	11.92±1.385 ^{Aa}	8.19±0.162 ^{Bbc}
10	34.24±0.091 ^{Ac}	32.24±0.021 ^{Bcdef}	1.09±0.017 ^{Bcd}	1.46±0.007 ^{Ac}	10.20±0.049 ^{Aa}	6.93±0.035 ^{Bcd}
11	35.86±0.021 ^{Ab}	34.75±0.007 ^{Bb}	1.36±0.000 ^{Bb}	1.89±0.010 ^{Ab}	12.62±0.063 ^{Aa}	10.82±0.021 ^{Bb}
12	32.61±0.898 ^{Ae}	32.54±0.565 ^{Acde}	1.36±0.141 ^{Bb}	1.91±0.056 ^{Ab}	11.75±3.521 ^{Aa}	10.76±2.489 ^{Bb}

*Different superscript letters in the same column indicate significant difference between mean values ($P < 0.01$). **Different superscript uppercase letters in the same row indicate significant difference between mean values ($P < 0.01$).

Results and discussion

Quality parameters

Free fatty acidity, peroxide and UV absorbance values of the olive oils extracted from Tavşan Yüreği cultivar in the Mobile Olive Oil Processing Unit (MOOPU) were shown in Table 1. Although a slight increase was observed during storage, all samples could be classified as extra virgin olive oils according to International Olive Oil Council (IOC) standards. Moisture content (results not shown) UF sample had higher (%0.09) moisture content than the filtered (%0.05) indicating filtration reduced moisture content.

A slight increase was observed in the free acidity values of EVOO during storage (Tab. 1). A similar trend has seen both F and UF samples suggesting that filtration had no detectable effect on free acidity values. It is well known that

free acidity increased with storage depending on the packaging material, storage conditions and time (Méndez and Falqué, 2007; Baiano et al., 2014; Abdalla et al., 2014; Lavelli et al., 2006; Clodoveo et al., 2007). Peroxide values (PV) was lower in the F samples than that of UF samples at the early stage of storage, after second months an increasing trend was observed in F samples (Tab. 1). The PV of UF samples started to increase in the fourth month. The PV reached maximum values and were comparable for F and UF samples near the end of storage. Significant increases were reported on the PV of olive oil samples during short term (30 days) and long term (sixth years) storage in different packaging materials at different conditions (Abdalla et al., 2014; Lavelli et al., 2006; Clodoveo et al., 2007; Oko-

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TABLE 3: Changes of tocopherol isomers contents in extra virgin olive oils extracted from Tavşan Yüreği during storage (ppm).

Storage period (month)	α-Tocopherol		β-Tocopherol		γ-Tocopherol	
	F	UF	F	UF	F	UF
0	307.63±4.606 ^{Aa}	302.70±7.968 ^{Ba}	1.96±0.014 ^{Aa}	1.45±0.012 ^{Ba}	1.62±0.001 ^{Aa}	1.21±0.006 ^{Ba}
2	295.50±0.694 ^{Ba}	298.86±6.789 ^{Aa}	0.94±0.018 ^{Ab}	0.98±0.002 ^{Ab}	1.45±0.005 ^{Ab}	1.01±0.002 ^{Bb}
4	280.28±10.056 ^{Ab}	274.45±0.357 ^{Bb}	0.85±0.001 ^{Ac}	0.83±0.008 ^{Ac}	1.02±0.007 ^{Ac}	0.83±0.006 ^{Bc}
6	260.96±1.199 ^{Bc}	260.22±9.961 ^{Abc}	0.77±0.007 ^{Ad}	0.53±0.013 ^{Bd}	0.88±0.002 ^{Ad}	0.65±0.009 ^{Bd}
8	233.17±11.124 ^{Bd}	242.51±0.707 ^{Ac}	0.43±0.015 ^{Ae}	0.48±0.014 ^{Ae}	0.66±0.006 ^{Ae}	0.43±0.007 ^{Be}
10	215.84±0.318 ^{Be}	221.37±15.155 ^{Ad}	0.41±0.004 ^{Ae}	0.33±0.013 ^{Bf}	0.31±0.006 ^{Af}	0.25±0.008 ^{Bf}
12	214.53±0.728 ^{Be}	129.60±8.092 ^{Be}	0.30±0.001 ^{Af}	0.29±0.001 ^{Ag}	0.20±0.003 ^{Ag}	0.15±0.010 ^{Bg}

*Different superscript letters in the same column indicate significant difference between mean values ($P < 0.01$). **Different superscript uppercase letters in the same row indicate significant difference between mean values ($P < 0.01$).

TABLE 4: Changes in total phenol contents (ppm) of extra virgin olive oil extracted from variety Tavşan Yüreği during storage (12 months).

Storage period (month)	Sample name	
	F	UF
0	164.87±3.740 ^{Ba}	179.45±0.297 ^{Aa}
1	149.41±0.063 ^{Bb}	175.39±0.827 ^{Ab}
2	145.98±0.325 ^{Bc}	165.88±0.516 ^{Ac}
3	139.30±0.544 ^{Bd}	143.38±4.193 ^{Ad}
4	137.53±0.445 ^{Bde}	139.11±1.216 ^{Ae}
5	136.45±0.282 ^{Ae}	135.53±0.665 ^{Bf}
6	130.39±0.233 ^{Bf}	132.92±0.559 ^{Agf}
7	129.15±0.841 ^{Bf}	131.36±0.714 ^{Ag}
8	125.73±0.679 ^{Bg}	128.05±0.283 ^{Ah}
9	123.21±0.007 ^{Bh}	124.40±0.212 ^{Ai}
10	120.17±0.050 ^{Ai}	120.30±0.098 ^{Ai}
11	119.14±0.091 ^{Ai}	118.75±0.714 ^{Ai}
12	118.12±0.162 ^{Ai}	118.11±0.014 ^{Ai}

*Different superscript letters in the same column indicate significant difference between mean values ($P < 0.01$). **Different superscript uppercase letters in the same row indicate significant difference between mean values ($P < 0.01$).

absorbance values fluctuation in olive oil storing studies (Méndez and Falqué, 2007; Baiano et al., 2014; Lavelli et al., 2006; Okogeri and Tasioula-Margari, 2002; Caponio et al., 2005; Gómez-Alonso et al., 2007; Del Caro et al., 2006).

geri and Tasioula-Margari, 2002). UV absorbance values (K_{232} and K_{270}) had decreasing and increasing trend storage time. Storage time and filtration had significant effects on UV absorbance values ($P < 0.01$). A fluctuation was seen in K_{232} values of F and UF samples and the changes on K_{232} values of UF sample had similar to that of filtered one. Decreases were observed in K_{270} value for F samples during early and late stages of the storage. The highest value was obtained in the eighth month. Higher K_{270} values indicated a sensitivity to oxidation for UF samples. These results are in agreement with the related literature about UV

of all samples during storage, generally, UF samples had lower L and b values indicating a dark green color for this oil. These changes have been attributed to the decomposition of color pigments such as chlorophylls, pheophytins, xanthophylls and carotenes (Boskou, 2006).

Tocopherol Profile

Tocopherol (α , β , γ) profiles of the EVOO were determined every two months during storage (Table 3). The results showed that tocopherol contents (α , β , γ) decreased with increasing storage time. The lowest tocopherols content was obtained at the end of storage. 30 % of α -tocopherol, 80 % of β -tocopherol and 88 % of γ -tocopherol contents were decomposed during storage. The amounts of tocopherols (α -tocopherol, β -tocopherol, γ -tocopherol) were higher in F samples. Tocopherol content of EVOOs changed significantly by storage time and filtration process ($P < 0.01$). Results are in agreement with Jabeur et al. (2016), while Lozano-Sanchez et al. (2012) and Jukic Spika et al. (2019) recorded no changes with applied filtration.

Total Polyphenolic Compounds

Total polyphenols contents of the samples were presented in Table 4. The highest total polyphenol values were determined at fresh oil which decreased with time. But the decreases were not as sharp as tocopherols, after one year 28.35 % and 34.18 % of total polyphenols were decomposed in F and UF samples, respectively. UF samples had higher total polyphenol content indicating that filtration had a negative effect on the total polyphenol in earlier months of storage time. There was a significant difference among EVOOs polyphenols in terms of storage time and filtration process ($P < 0.01$). After a short term or long term

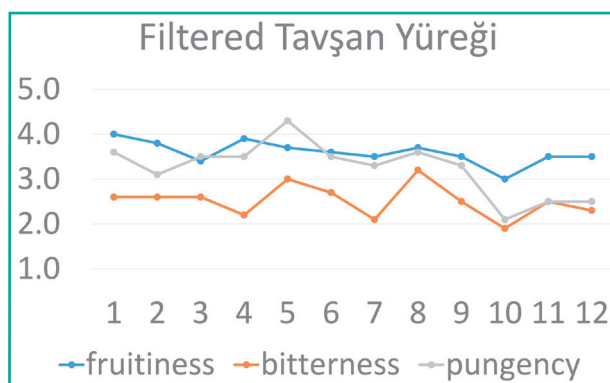


FIGURE 2: Sensory values of filtered Tavşan Yüreği (Antalya) olive oils during 12 months of storage.

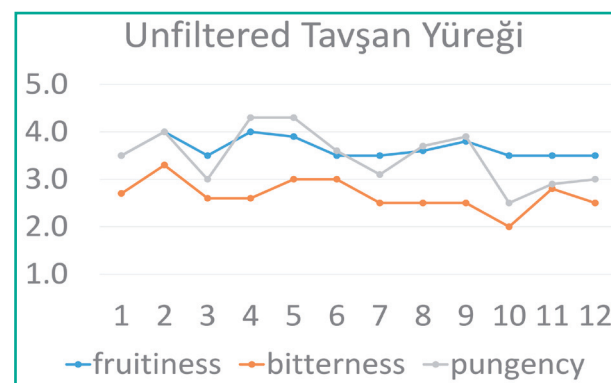


FIGURE 3: Sensory values of unfiltered Tavşan Yüreği (Antalya) olive oils during 12 months of storage.

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storage significant decreases in total polyphenols were reported for monocultivar and commercial olive oils by Clodoveo et al. (2007), Morelló et al. (2004), Abdalla et al. (2014) and Baiano et al. (2014).

Phenolic Profiles

Phenolic profiles of the samples were determined monthly (Tab. 5 and 6). Phenolic profiles of filtered EVOO are shown in Table 5. Luteolin was the most abundant phenolic that has been identified for all samples. Although most of the phenolics content decreased with storage, 4-hydroxy phenyl acetic acid increased with storage. Tyrosol was detected only in fresh (9.17 ppm) and stored olive oil after four months (8.66 ppm). Up to fifth month 4-hydroxy benzoic acid increased and then decreased. Fresh olive oil had syringic acid but it was not detected in stored samples. Vanillin was identified at the of storage. Trans ferulic acid appeared after four months and it decreased (0.45–0.02 ppm) with storage. m-coumaric acid content decreased at the early stage of storage then increased after six months. Amount of m-coumaric acid decreased

at the end of storing. A similar trend was observed for oleuropein and the highest content (6.92±0.97) was detected in fifth months.

UF samples had a wider range of phenolic compounds (Tab. 6). Tyrosol content of UF sample increased up to four months then decreased. After eight month an increa-

TABLE 5: Changes in phenolic compounds of filtered Tavşan Yüreği (Antalya) during 12 months of storage time (ppm).

Phenolic compounds	Months												
	0	1	2	3	4	5	6	7	8	9	10	11	12
tyrosol	9.17±0.187	nd	nd	nd	8.66±0.147	nd	nd	nd	nd	nd	nd	nd	nd
4-hydroxy benzoic acid	3.12±0.120	3.32±0.177	4.39±0.048	3.70±0.243	1.19±0.057	7.68±0.640	7.88±0.317	8.50±0.226	7.74±0.025	7.60±0.075	6.33±0.413	3.96±0.612	2.50±0.771
4-hydroxy phenyl acetic acid	nd	nd	nd	0.62±0.067	nd	nd	0.39±0.052	nd	nd	nd	nd	nd	7.48±0.128
syringic acid	1.08±0.152	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd
vanillin	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.78±0.051
trans-ferulic acid	nd	nd	nd	nd	nd	0.45±0.055	0.32±0.067	0.12±0.027	0.08±0.032	0.07±0.021	0.05±0.012	0.04±0.011	0.02±0.013
m-coumaric acid	1.19±0.023	0.94±0.081	0.71±0.026	1.05±0.051	1.25±0.213	0.86±0.144	1.40±0.123	1.94±0.121	1.84±0.168	1.50±0.115	1.40±0.019	1.26±0.023	0.72±0.069
oleuropein	3.15±0.213	3.19±0.127	3.21±0.032	3.25±0.534	3.31±0.317	6.92±0.967	6.25±0.624	5.51±0.049	5.02±0.257	3.33±0.034	2.96±0.177	2.36±0.051	2.01±0.222
luteolin	130.56±2.438	130.11±1.121	125.68±3.615	123.76±1.156	120.00±2.156	114.72±3.849	111.68±1.012	108.24±2.014	106.67±0.262	100.19±4.292	94.81±3.629	92.22±2.814	87.08±3.714
apigenin	2.61±0.122	2.22±0.218	1.97±0.026	1.94±0.027	1.90±0.217	1.88±0.027	1.65±0.067	1.53±0.014	1.47±0.014	1.31±0.014	1.07±0.064	0.63±0.028	0.30±0.002

nd: not detected; N = 16

TABLE 6: Changes in phenolic compounds of unfiltered Tavşan Yüreği (Antalya) during 12 months of storage time (ppm).

Phenolic compounds	Months												
	0	1	2	3	4	5	6	7	8	9	10	11	12
tyrosol	0.57±0.019	1.23±0.028	3.62±0.299	4.12±0.427	14.36±1.166	7.32±0.047	6.23±0.067	5.23±0.221	4.38±0.235	16.74±0.679	12.49±0.418	10.23±0.628	16.38±3.033
4-hydroxy benzoic acid	5.18±0.422	4.02±0.237	3.49±0.078	3.16±0.271	0.31±0.013	0.20±0.011	4.19±0.812	8.23±1.026	8.51±1.124	0.43±0.014	6.35±1.097	8.31±0.736	7.78±0.345
4-hydroxy phenyl acetic acid	3.11±0.615	1.17±0.178	0.79±0.056	0.66±0.012	nd	nd	nd	nd	nd	nd	nd	nd	nd
syringic acid	nd	nd	nd	nd	1.66±0.041	nd	nd	nd	nd	2.30±0.137	nd	nd	nd
vanillin	0.96±0.023	0.85±0.027	0.81±0.012	0.77±0.037	0.75±0.047	0.71±0.032	0.66±0.047	0.56±0.057	0.51±0.048	0.48±0.058	0.41±0.066	0.20±0.022	0.11±0.072
p-coumaric acid	nd	nd	nd	nd	nd	nd	nd	nd	nd	0.09±0.001	nd	nd	nd
trans-ferulic acid	0.12±0.001	nd	nd	nd	nd	0.11±0.019	nd	0.01±0.000	0.05±0.025	nd	nd	nd	nd
m-coumaric acid	3.51±0.562	1.54±0.112	1.56±0.010	1.66±0.011	2.1±0.291	3.02±0.421	1.43±0.028	1.19±0.013	3.15±0.446	3.28±0.111	1.40±0.017	1.23±0.042	1.12±0.019
o-coumaric acid	0.12±0.016	0.12±0.012	0.12±0.013	0.14±0.011	0.15±0.021	0.08±0.002	0.06±0.001	0.06±0.007	0.05±0.001	0.04±0.000	0.03±0.001	0.02±0.001	0.01±0.000
oleuropein	2.06±0.087	2.14±0.314	3.11±0.214	1.49±0.080	1.02±0.115	1.23±0.014	1.67±0.021	2.16±0.029	2.19±0.057	3.63±0.061	2.25±0.056	2.63±0.082	1.30±0.014
luteolin	151.37±4.462	136.36±5.177	126.17±6.077	122.89±2.721	110.68±8.608	104.73±3.679	104.08±1.042	103.38±0.233	103.07±0.564	85.88±10.523	83.35±1.531	81.83±1.104	75.41±3.240
apigenin	5.08±0.357	4.81±0.219	4.72±0.210	3.33±0.521	2.41±0.214	2.21±0.207	2.10±0.114	1.78±0.035	1.24±0.021	0.69±0.014	0.55±0.026	0.42±0.024	0.29±0.121

nd: not detected; N = 16

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sing trend started and reached maximum at the end of storage (16.38 ppm). 4-hydroxy benzoic acid showed a similar trend. 4-hydroxy phenyl acetic acid was detected up to first three months with a decreasing trend. Syringic acid was detected only fourth and ninth months and *p*-coumaric acid was identified in ninth month. Vanillin and trans ferulic acid contents decreased during storage (0.96 to 0.11 ppm). *m*-coumaric acid content fluctuated with storage. Lower oleuropein, luteolin and apigenin contents were determined in samples stored for twelve months. These results showed that filtration caused changes on the phenolic profile. Yorulmaz et al. (2009) reported that luteolin was the most abundant phenolic compound following trans-cinnamic acid and luteolin-7-glucoside in Turkish EVOO. They also quantified tyrosol, syringic acid, *p*-coumaric acid, luteolin-7-glucoside, trans cinnamic acid, luteolin and apigenin in olive oils extracted from different Turkish varieties. Morrelló et al. (2004) noted that storage did not have any effect on vanilic acid or vanillin, (which were present at low concentration) there was a significant decrease in the concentration of other quantified phenolic compounds. Among the most representative phenolic compounds in olive oil, lignans seem to be the most stable during oil storage. Mulinacci et al. (2013), Gómez-Alonso et al. (2007) and García et al. (2003) showed an increase of tyrosol and hydroxytyrosol contents over time due to hydrolytic processes of the secoiridoidic derivatives. Baiano et al. (2014) reported that there were increasing and decreasing trends in phenolic compounds (3,4-DHPEA, *p*-HPEA, vanillin, *p*-coumaric acid, 3,4-DHPEA-AC, 3,4-DHPEA-EDA, *p*-HPEA-AC, *p*-HPEA-EDA, 1-acetoxipinoresinol + *trans*-cinnamic acid, *p*-HPEA-EA) content. Phenolic alcohols such as hydroxytyrosol and tyrosol are present in fresh virgin olive oils at relatively low concentrations, but their amount is increased after storage, due to hydrolysis of secoiridoids such as dialdehydic form of elenolic acid linked to hydroxytyrosol (3,4-DHPEAEDA) or tyrosol (*p*-HPEA-EDA) and an isomer of the oleuropein aglycon (3,4-DHPEA-EA) and hydroxytyrosol acetate (3,4-DHPEA-AC) indicating a more active participation in the oxidative processes as they were more easily oxidized (Angerosa et al. 1995; Cinquanta et al. 1997; Mulinacci et al., 2013). The formation of simple phenols in present study due to the hydrolysis of their secoiridoid derivatives was also greater in UF samples (22.89 ppm and 20.46 ppm in UF and F samples, respectively, after 9 months of storage). The main reason for this effect could be attributed to the filtration process.

Sensory Analysis

Tavşan Yüreği extra virgin olive oil has a green fruity aroma characterized by caramel, honey, blossom, green apple, grass, freshly cut grass, almond and spring notes. It has no-defect and fruitiness is higher than bitterness and pungency (Fig. 2 and 3). Fruitiness was protected the whole year and pungency decreased at the end of the storage time. Fruitiness and bitterness were higher even in unfiltered conditions. During storage of samples, fruitiness has been reduced from 4 to 3.5 for both filtered and unfiltered. Filtered and unfiltered EVOO samples had bitterness score between 3 and 2 during storage time. However, pungency was 3.8 out of 10 in the beginning and reduced to 2.5 in filtered EVOO samples. This value was 3.5 in unfiltered and decreased to 3 at the end of storage time. Tasting panel did not detect any defects in the samples during the whole 12 months' storage in room temperature. This can be attributed to high-quality and good storage conditions of EVOO.

Conclusion

The results obtained in this study showed that Tavşan Yüreği olive oils have a unique chemical composition and good oxidative stability due to suitable tocopherols and phenolics contents. Filtration process decreased total polyphenol and phenolic compounds concentration in EVOO samples. At the end of the storage time, 28.35 % and 34.18 % of total polyphenols were decomposed in F and UF samples, respectively. Luteolin was the most abundant phenolic component that has been identified in Tavşan Yüreği olive oils. UF samples showed more identified and higher phenolic content during storing. On the other hand, 30 % of α -tocopherol, 80 % of β -tocopherol and 88 % of γ -tocopherol contents were decomposed during a year of storage period. The filtration process had no significant effect on oxidation stability parameters (free fatty acidity, peroxide and UV absorbance values), and it led to the higher lightness of filtered EVOO samples. Color of EVOO samples changed from green to yellow after 12 months' storage. Oxidative stability parameters showed a slight increase during storage and EVOO samples' free fatty acidity, peroxide and UV absorbance values were under the limitation of IOC regulation. The data provided could make a contribution to extent of geographic indications in Turkish olive oils.

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Conflict of interest

The authors have declared no conflict of interest.

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