EFFECTS OF FILTRATION PROCESS AND STORAGE TIME ON THE CHEMICAL CHANGES AND SENSORY PROPERTIES OF OLIVE OIL EXTRACTED FROM TURKISH USLU CULTIVAR

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Abstract. Upper Mesopotamia is a part of Turkish territory is the homeland of the olive tree with a wide range genetic resource. This is the first report on chemical composition and oxidative stability of olive oil extracted from Uslu cultivar grown locally in a small amount. In this research, a Turkish olive cultivar named as "Uslu" locally grown in Akhisar was used for production of monocultivar extra virgin olive oil by using Mobile Olive Oil Processing Unit". Olive oil samples were bottled before and after filtration and stored up to 24 months. Some chemical properties such as free fatty acid content, peroxide value, moisture content, UV absorbance value, minor and major components (fatty acid composition, tocopherols, total phenol compounds and phenolic composition), were determined during storage for 24 months. Chemical parameters such as free fatty acid, peroxide value except UV absorption values of both filtered and unfiltered "Uslu" olive oil samples were in agreement with the trade standards of International Olive Council (IOC). Color values of EVOO changed from green to yellow while UV absorbance values altered during storage. Very low free fatty acidy (0.2%) values which are unusual for commercial olive oils in Turkey were obtained for filtered and unfiltered samples. A slight increase was seen for unfiltered sample at the end of storage. Filtration had no detectable effect on fatty acid profile. Filtered sample had higher total phenols (407.64±4.051 ppm) and α -tocopherol (237 and 123.31 ppm) contents than unfiltered ones and their contents decreased approximately 50% at the end of storage. Luteolin was the most abundant phenolic compound and its concentration decreased from 268.65±5.428 to 93.57±0.541ppm during storage. It seemed effect of filtration was more obvious on total phenolic contents. This study was good practice for producing premium extra virgin olive oil by using Mobile Olive Oil Processing Unit. The results obtained in this study showed that Uslu olive oils has a unique chemical composition and a good oxidative stability with high tocopherols and phenolics contents that are uncommon in most of the commercial olive oils.

Key words: Olive oil, Uslu, Phenolic Compounds, Tocopherol, Storage.

Abbreviation: F= filtered, UF= unfiltered, EVOO= extra virgin olive oil, VOO= Virgin Olive Oil.

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Introduction. Formulation of the problem

Olive is one of the most important plant in Mediterranean countries, especially Spain, Italy, Greece and Turkey. Uslu is a domestic variety in the Manisa district of the Middle Aegean zone and it is generally used for black table olive production due to its large shape, soft texture with low olive content (18-20%). An abundance of oleic acid, a monounsaturated fatty acid, linoleic and linolenic acids as polyunsaturated fatty acids, are the characteristics that sets olive oil apart from other vegetable oils. From the ancient times, people of Mediterranean countries consume extra virgin olive oil (EVOO), because olive oil is unique oil among all edible oils due to high amounts of phenolics, vitamins, oleic acid and other minor compounds. The chemical composition varies depending on the genetic, geographic, agronomic processing and storage conditions. Its shelf life longer than other edible oils, because of presence of antioxidants such as mainly polar phenols and atocopherol. Other factors such as free fatty acids, unsaturated hydrocarbons, enzymes, and trace metals are affected oxidative stability negatively. Pigments have negative effect on oxidative stability. EVOO's major and minor components as well as oxidation indices of virgin olive oil were changed during storage.

Analysis of recent research and publications

Oxidative stability parameters such as, free fatty acidy, peroxide value and oxidative rancidity increased during storing time. Total polyphenols declined up to73%, and this decrease was remarkable higher in samples whose initial phenol contents were greater. Another important factor in olive oil quality is storage conditions. Storage at room temperatures led to no change in the amount of some phenolics such as tyrosol and hydroxy tyrosol. Storage of olive oil under nitrogen pressure in a dark place at room temperature (25-30°C or lower) increases shelf life [1]. There was no change in aromatic hydrocarbons of freezed samples up to 12 months [2] and no significant changes were observed in the unsaturated fatty acid composition [3,4]. Important reduction (79%) was observed in the amounts of α tocopherol (Vitamin E) in four months, whereas <45% of the phenols were lost under diffused light during storage [5]. A positive correlation was observed between the age of the oils and the tyrosol to total phenols ratio [6]. EVOO protects its premium quality after 240 days of storage at 40°C due to high antioxidant content [7]. A decrease in chlorophyll and carotenoid contents and an increase of oleic acid percentage were also reported [8]. Psomiadou et al. [9] suggested a good handling is quite important for retaining high α tocopherol levels of Greek VOO under domestic conditions up to two years. Filtration process of EVOO showed a gradual loss in stability during the storage due to a lower total phenolic content [10]. Especially cotton filter caused to a significant loss in the amount of hydroxy tyrosol in the laboratory scale [11]. Fregapane et al. [12] reported that filtration and especially dehydration could help prolong the shelf life of some high quality olive oils but less stable virgin olive oils. Although some physicochemical characteristics of Uslu EVOO such as free fatty acid, iodine value, peroxide value, saponification, unsaponifiable matter, refractive index and specific gravity values were reported earlier, this is the first report on monitoring the changes of Uslu quality during shelf life in details [13]. A mobile olive oil processing unit (MOOPU) was designed to produce "monovarietal virgin olive oil" with premium quality. MOOPU was transferred into the orchard located in Manisa district of the Middle Aegean zone. Therefore it was possible to process Uslu olives at optimum conditions within two hours after harvest. Olive oils were packaged before and after filtration and quality parameters were determined and monitored during storage monthly for 2 years.

The aim of the present study was processing of the olives that are harvested in their own ecological environment within a couple of hours and producing of EVOO with premium quality. Uslu EVOO quality and economic potential have not been explored up to date due to mishandling during processes from the garden to table.

Research materials and methods

Production of Extra Virgin Olive Oil (EVOO). A "Mobile Olive Oil Processing Unit" (MOOPU) with state-of-the art Olemio equipments was designed in order to produce premium quality EVOO (Fig. 1).

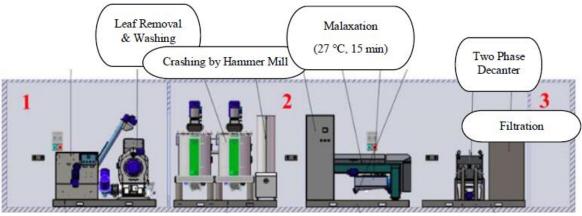


Fig. 1. Mobile olive oil processing unit (mill on wheel)

A special container was constructed and 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 243 x12 192x2896 mm which is divided into three separate sections. First section is olive accepting unit including; bunker, leaf removers, washer and crusher units of the system. Second section is processing unit including malaxer, decanter, filter and bag-in-box filling machine. 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. MOOPU carried by a trailer truck to orchards in 2014-2015 season. Olive fruits were harvested by hand picking in the early harvest period and processed to "cold press" EVOO in the MOOPU in a few hours. Olive paste was prepared after crushing by a hammer mill and the paste was mixed in the malaxer at 27°C for 15 min (Cold press). After decantation EVOO were packaged before (Unfiltered)

and after filtration (Filtered). A filter press (Oliomio Jolly 40, Italy) with the paper (Gruppo Cardenons E2, paper weight: 350 g/m^2 , 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: 4cm) and filled by nitrogen gas. The bottles were stored at room temperature (18-24°C) up to 24 months and analyzed monthly.

Chemical analyses. Free fatty acid content, peroxide value, moisture were performed according to the EC 2598/9 [14], The American Oil Chemists' Society Cd 8-53 methods [15], ISO 662 [16] respectively. Color values (*L*, *a*, *b* values) were determined by spectrophotometer (Minolta, CM-3600d, Japan). *L* (lightness), *b* (yellowness), and *a* (redness) values were measured. UV absorbance was measured at 232, 266, 270 and 274 nm by using UV Spectrophotometer (Agilent 8453, USA) according the International Olive Council method COI/T.20/Doc. No 19/Rev. 3. [17]. Δ K values were calculated with the following formula:

$\Delta K = K_{270} - [(K_{266} + K_{274})/2]$

Fatty Acid Composition. Fatty acid composition was performed according to the method of IOC [18]. Analysis was performed by the TRACE[™] Ultra Gas Chromatograph equipment (Thermo Fisher Scientific, USA) with the following operation conditions. The TRACETM Ultra Gas Chromatograph equipped with Flame ionization detector, split injector (40:1), HP-88 column (100-meter length, 0.25 mm I.D, 0.20 µm film thickness) were used for separation. The carrier gas was Helium (with 1mL/min flow rate), and initial temperature was 100°C. The temperature ramping rate was 4°C/min. Injection temperature and detector temperature were 240°C and 250°C respectively.

Total Phenolic Content. Polar fraction was extracted and used for total phenolic and phenolic composition analyses [19]. Olive oil sample (2.5 g) 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 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 µm microfilter (Merck, PVDF, Millipore Millex-HV, Germany) and poured in to 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. Phenolic standars (apigenin, cafeic acid, gallic acid, luteolin, m-cumaric acid, p-coumaric acid, oleuropein, syringic acid, trans-ferulic acid, vanilic acid, vanillin, tyrosol, 3-hydroxy tyrosol, 3.4-dihydroxy benzoic acid, 4-hydroxy benzoic acid, 4-hydroxy phenyl acetic acid) were purchased from Sigma-Aldrich.

Tocopherol Composition. Tocopherol composition was performed using AOCS Official Method Ce 8-89 [20]. 2 g EVOO sample was weighed into a 25 mL volumetric flask. After dissolving oil by a quantity of hexane, flask was made up to volume. Solution was passed from syringe filter (0.45 μ m) (PVDF, Millipore Millex-HV) in to 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 × 250 mm length and 5 µm particle size) was used for analysis. Column temperature was fixed at 30°C during process. Flow rate of analysis was 1 mL/min. Isopropanol: hexane (0.5:99.5, v/v) isocratic mix was used for mobile phase, and chromatograms were obtained at 292 nm wavelength. Analysis time and injection volume were 30 min and 100 µl, respectively. Tocopherol standards were purchased from Sigma-Aldrich and used for determination of α , β , γ and Δ tocopherols contents.

Sensory Analysis. Every month olive oil samples were evaluated 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 [21]. Eight trained tasting 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 storaging, 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.

Results of the research and their discussion

Chemical Analyses. Free acidity, peroxide and UV absorbance values of the olive oils extracted from Uslu variety in the Mobile Olive Oil Processing Unit (MOOPU) were shown in Table1. Although a slight increase were observed in the free fatty acid values during storage, all samples could be classified as extra virgin olive oils according to International Olive Oil Council (IOC) standards. Moisture content of filtered and unfiltered samples were comparable indicating filtration had detectable effect on the moisture content. Unfiltered EVOO of Uslu (Manisa) had higher moisture content of EVOOs was under the limitation of International Olive Oil Council (<%0.2).

Free fatty acid content of filtered and unfiltered EVOO samples of Uslu (Manisa) showed same trend during first year of storage time (Table 1), but unfiltered type showed increase in thirteenth month. The results showed that free acidity of EVOOs had significant differences during 2-year storage period (P>0.01). Some researchers showed that free acidity increased with storage depending on the packaging material, storage conditions and time [4,7,22-24]. According to the results filtration showed detectable effect on free fatty acid content after two years storing.

Peroxide values (PV) of early stage of oxidation was higher in the filtered samples than that of unfiltered samples at the beginning of storage, after third month both filtered and unfiltered showed same trend up to end of storage time (Table 1). The PV reached to maximum values and were comparable for filtered and unfiltered samples at the beginning of storage (P<0.01). Significant increases were reported on the PV of olive oil samples during short term (30 days) and long term (sixth years) of storage in different packaging materials at different conditions [5,7,23].

Storage	Free Fa	tty Acid	Peroxide	Value,	K'	232	K2	270
Period	Conte	nt (%)	meq O2	/Kg oil	K2	-52	K 2	470
(Month)	F	UF	F	UF	F	UF	F	UF
0	0.1 ± 0.00^{b}	0.1±0.00 ^c	2.97±0.000 ^j	5.96±0.007°	1.7 ± 0.00^{i}	1.8 ± 0.00^{i}	0.10 ± 0.000^{j}	0.12 ± 0.000^{1}
1	0.1 ± 0.00^{b}	0.1±0.00 ^c	11.93±0.007 ^a	5.99±0.030°	1.8±0.00 ^h	1.7 ± 0.00^{j}	0.21±0.000b	0.21±0.000°
2	0.1 ± 0.00^{b}	0.1±0.00 ^c	11.90±0.037 ^a	8.22±0.038 ^a	1.7 ± 0.00^{i}	1.7 ± 0.00^{j}	0.14 ± 0.000^{f}	0.14±0.000 ^j
3	0.1 ± 0.00^{b}	0.2 ± 0.00^{b}	8.94±0.037 ^{bcd}	8.98±0.012 ^b	1.6 ± 0.00^{j}	1.6 ± 0.00^{k}	0.07 ± 0.000^{m}	0.10±0.000 ⁿ
4	0.1 ± 0.00^{b}	0.2 ± 0.00^{b}	8.91 ± 0.002^{df}	8.97±0.030 ^b	1.6±0.00 ^j	1.5 ± 0.00^{1}	0.09 ± 0.000^{k}	0.12 ± 0.000^{1}
5	0.2 ± 0.00^{a}	0.2 ± 0.00^{b}	8.90 ± 0.004^{df}	8.97±0.018 ^b	2.2±0.00 ^d	2.0±0.00g	0.21±0.000b	0.14±0.000 ^j
6	0.2 ± 0.00^{a}	0.2±0.00 ^b	8.90 ± 0.005^{df}	8.97±0.002 ^b	2.1±0.00 ^e	2.0±0.00g	0.21±0.000b	0.09±0.000°
7	0.2 ± 0.00^{a}	0.2 ± 0.00^{b}	8.93±0.002 ^{cd}	8.97±0.007 ^b	2.1±0.00 ^e	2.1±0.00 ^f	0.12±0.000 ^h	0.19±0.000 ^d
8	0.2 ± 0.00^{a}	0.2 ± 0.00^{b}	8.99±0.005 ^b	8.98±0.012 ^b	2.3±0.00°	0.0 ± 0.00^{q}	0.21±0.000b	0.18±0.000e
9	0.2 ± 0.00^{a}	0.2 ± 0.00^{b}	8.99±0.010bc	8.98±0.004 ^b	1.9±0.00g	1.7±0.00 ^j	0.17 ± 0.000^{d}	0.16±0.000g
10	0.2 ± 0.00^{a}	0.2±0.00 ^b	8.99±0.005 ^b	8.98±0.021 ^b	2.1±0.00 ^e	2.2±0.00 ^e	0.17 ± 0.000^{d}	0.18±0.000e
11	0.2 ± 0.00^{a}	0.2 ± 0.00^{b}	8.97±0.028 ^{bc}	8.90±0.006 ^b	2.1±0.00 ^e	1.9±0.00 ^h	0.21±0.000 ^b	0.15±0.000 ^h
12	0.2 ± 0.00^{a}	0.2 ± 0.00^{b}	8.95±0.057 ^{bcd}	8.89±0.005 ^b	1.9±0.00 ^f	2.0±0.00g	0.05±0.000 ⁿ	0.13±0.000 ^k
13	0.2 ± 0.00^{a}	0.2 ± 0.00^{b}	8.95±0.022 ^{bcd}	8.85 ± 0.008^{b}	1.9±0.00g	2.0±0.00g	0.05±0.000 ⁿ	0.13±0.000 ^k
14	0.2±0.01 ^a	0.3±0.00 ^a	8.88 ± 0.025^{f}	8.81±0.005 ^b	0.4±0.00 ⁿ	$0.5\pm0.00^{\circ}$	-0.25±0.000°	-0.17±0.000 ^p
15	0.2±0.01 ^a	0.3±0.00 ^a	8.88 ± 0.003^{f}	8.81±0.002 ^b	1.3±0.00 ^k	0.3±0.00 ^p	0.36±0.000 ^a	0.31±0.000 ^b
16	0.2±0.01 ^a	0.3±0.00 ^a	8.66±0.022 ^e	8.75±0.012 ^b	0.8 ± 0.00^{m}	0.8±0.00 ⁿ	0.11±0.000i	0.17 ± 0.000^{f}
17	0.2 ± 0.00^{a}	0.3±0.00 ^a	8.66±0.005e	8.68±0.011 ^b	1.9±0.00g	2.0±0.00g	0.08 ± 0.000^{1}	0.09±0.000°
18	0.2 ± 0.00^{a}	0.3±0.00 ^a	8.57 ± 0.002^{f}	8.59±0.006 ^b	0.9 ± 0.00^{i}	1.1±0.00 ^m	0.08 ± 0.000^{1}	0.15±0.000 ^h
19	0.2 ± 0.00^{a}	0.3±0.00 ^a	8.48±0.022g	8.57±0.002 ^b	2.2±0.00 ^d	3.0±0.00 ^a	0.15±0.000e	0.21±0.000°
20	0.2±0.01 ^a	0.3±0.00 ^a	8.45±0.016g	8.52±0.012 ^b	2.3±0.00°	2.4±0.00°	0.14 ± 0.000^{f}	0.16±0.000g
21	0.2±0.01ª	0.3±0.00 ^a	8.45±0.011g	8.48±0.013 ^b	2.8±0.00 ^a	2.4±0.00°	0.14 ± 0.000^{f}	0.17 ± 0.000^{f}
22	0.2 ± 0.00^{a}	0.3±0.00 ^a	8.32±0.006 ^h	8.27±0.025 ^b	2.4±0.00 ^b	2.5±0.00 ^b	0.19±0.000°	0.34±0.000 ^a
23	0.2 ± 0.00^{a}	0.3±0.00 ^a	8.32 ± 0.002^{h}	8.25±0.021b	2.4±0.00 ^b	2.2±0.00 ^e	0.13±0.000g	0.11±0.000 ^m
24	0.2 ± 0.00^{a}	0.3±0.00 ^a	8.12 ± 0.005^{i}	8.14±0.006 ^b	2.4±0.00 ^b	2.3±0.00 ^d	0.13±0.000g	0.14±0.000 ⁱ
*Differe	ent superscript	letters in the sa	me column indicate	significant differ	ence between n	nean values (P <	(0.01).	

Table 1 – Oxidative stability parameters of extra virgin olive oils during 24 months storage

UV absorbance values (K232 and K270) which are indicator of oxidation changed during storage significantly. K232 value of filtered Uslu (Manisa) EVOO decreased up to fourth month. There was an increase in fifth and eighth months. The minimum and maximum level of K232 value obtained in fourteenth month and twenty-first month, respectively. It decreased in near the end of storage time. The changes on K232 values of unfiltered Uslu (Manisa) EVOOs had similar pattern to the those of filtered one. At early stage of storage, it decreased and increased between fifth and seventh months. A sharp decrease was seen in eighth month. The minimum and maximum level of K₂₃₂ value had also determined for unfiltered eighth and ninteenth month, respectively. According to the IOC standart K_{270} must be <0.22 for EVOO. Generally, there were significant differences among all EVOOs during storage (P<0.01). Filtered Uslu (Manisa) EVOO had the highest and the lowest value of K270 values in fourteenth and fifthteenth months, respectively (Table 1). Unfiltered Uslu (Manisa) EVOO had the highest K270 value in twentysecond month and the lowest was in fourteenth month. Δk values of filtered and unfiltered samples were zero or below zero (results are not shown). These results are in agreement in the related literature [4,7,22, 23, 25-27]. Baiano et al. [22] reported that K232 value of Coratina olive oil increased up to sixth year, then it decreased, at he end of final storage an increase was observed. Gutierrez and Fernandez. [28] showed that only two quality indices (K270 and sensory evaluation) Picual and Hojiblanca olive oils decreased during storage at 2°C in darkness and 30°C in illimunation. Quality deterioration resulted in downgraded olive oils which was no longer extra virgin olive oils during storage and there was an excellent correlation between initial stability and the time to reach the limit of K270>0.25.

Color Analysis. In spite of the fact that color is not regarded as an important quality characteristic for extra virgin olive oils, it has a great effect on consumer acceptance. Color of virgin olive oils is related to olive maturity and process conditions. Analysis of color (L, a and b values) showed that color of olive oil samples altered significantly during storage (Table 2). It has been attributed that decomposition of color pigments such as chlorophylls, pheophytins, xanthophylls and carotenes [1]. The lowest L values (lightness) were seen in twenty first month and seventh months for filtered and unfiltered samples

respectively. The highest L values were observed in eighth month for filtered and unfiltered samples. Generally, unfiltered samples had lower L and b values indicating they are dark green. Fluctuations were observed in a (redness)

and b (yellowness) values of all samples during storage. The highest b value were obtained for eighth month. After this month there was a decreasing trend in b values of both filtered and unfiltered samples.

Table 2 – Color values (l. a. b values) of filtered and unfiltered Uslu during 24 months of storage period

Storag Period	L va	alue	a v	alue	b va	lue
(Month)	F	UF	F	UF	F	UF
0	36.25±0.084 ^h	36.61±1.237 ^{fghi}	-1.6±0.247 ⁱ	-0.96±0.190efghi	10.83±1.845 ^{bcde}	12.83±1.470 ^{bcd}
1	36.77±1.668 ^{fgh}	35.61±0.919 ^{hij}	-0.86±0.028de	-0.90±0.109defgh	12.12±0.551bc	11.25±4.942 ^{cd}
2	36.37±0.565 ^h	38.75±0.784 ^b	-0.41±0.060 ^a	-0.24±0.070 ^a	14.86±0.438 ^a	14.85±0.905 ab
3	36.50±0.070gh	36.70±1.244 ^{efgh}	-0.61±0.116 ^{bc}	-0.59±0.014 ^{bc}	12.79±3.231b	13.44±2.283bc
4	38.15±0.141 ^{bcd}	37.73±0.282 ^{bcdef}	-0.90±0.010 ^{def}	-0.83±0.045 ^{def}	11.98±0.049 ^{bc}	12.22±0.494 ^{bcd}
5	37.06±0.685 ^{efgh}	38.00±0.622 ^{bcd}	$-0.88 \pm 0.014^{\text{def}}$	-0.97±0.003 ^{efghi}	11.78±1.011bc	11.52±0.593 cd
6	37.56±0.169 ^{cdef}	37.86±0.084 ^{bcd}	-0.1±0.017 ^{efg}	-1.04±0.014 ^{fghij}	11.76±0.466 ^{bc}	11.79±0.346 ^{bcd}
7	37.86±0.120 ^{bcde}	31.68±0.042 ^k	0.95±0.010 ^{efg}	-0.19±0.113 ^a	12.32±1.237bc	12.80±1.619 ^{bcd}
8	48.89±0.155 ^a	48.90±0.056 ^a	-1.21±0.007 ^h	-1.26±0.003 ^j	16.43±0.360 ^a	16.57±0.226ª
9	32.66±0.226 ^j	35.59±0.784 ^{ij}	-0.62±0.045bc	-0.50±0.084 ^b	11.09±1.400 ^{bcde}	10.94±0.106 ^{cde}
10	36.35±0.289 ^h	36.36±0.077 ^{ghij}	-0.92±0.035 ^{defg}	-0.92±0.038 ^{defgh}	9.26±0.565e	9.70±0.367 ^{de}
11	38.32±0.014 ^{bc}	38.46±0.042 ^{bc}	-0.94±0.003 ^{efg}	-0.95±0.010 ^{efgh}	11.48±0.007 ^{bcd}	12.30±0.014 ^{bcd}
12	33.60±0.219 ⁱ	35.76±0.134 ^{hij}	-0.58±0.045 ^{abc}	-0.87±0.09 ^{defg}	11.68±0.509 ^{bc}	11.31±2.128 ^{cd}
13	33.10±0.488 ^{ij}	35.26±0.573 ^j	-1.6±0.247 ^{ab}	-0.96±0.190efghi	12.18±0.198bc	12.81±0.007bcd
14	33.75±0.007 ⁱ	35.71±0.064 ^{hij}	-0.86±0.028bc	-0.90±0.109efghi	12.18±0.198bc	12.68±0.191bcd
15	37.63±0.007 ^{bcdef}	37.69±0.071 ^{bcdef}	-0.41±0.060 ^{gh}	-0.24±0.070 ^{ij}	10.96±0.007 ^{bcde}	11.34±0.057 ^{cd}
16	37.53±0.078 ^{cdef}	37.52±0.021 ^{cdef}	-0.61±0.116 ^{efg}	-0.59±0.014 ^{ghij}	10.86±0.212 ^{bcde}	11.05±0.014 ^{cd}
17	37.36±0.014 ^{defg}	37.42±0.085 ^{cdefg}	-0.90±0.010 ^{fgh}	-0.83±0.045 ^{hij}	10.66±0.057 ^{bcde}	10.98±0.092 ^{cde}
18	37.28±0.163 ^{defg}	37.41±0.021 ^{cdefg}	-0.88±0.014 ^{gh}	-0.97±0.003 ^{hij}	10.45±0.311 ^{cde}	10.97±0.014 ^{cde}
19	38.56±0.424 ^b	38.42±0.403bc	-0.1±0.017 ^{defg}	-1.04±0.014 ^{efgh}	12.27±0.339bc	12.10±0.226 ^{bcd}
20	37.99±0.092 ^{bcde}	37.81±0.792 ^{bcde}	0.95±0.010 ^{cd}	-0.19±0.113 ^{cde}	11.68±0.078 ^{bc}	11.87±0.813 ^{bcd}
21	31.77±2.432 ^k	32.38±3.861 ^k	-1.21±0.007 ^{defg}	-1.26±0.003b	9.51±2.659de	8.00±1.004 ^e
22	37.82±0.361 ^{bcde}	37.33±0.000 ^{cdefg}	-0.62±0.045de	-0.50±0.084 ^{defg}	11.33±0.636 ^{bcde}	12.34±1.506 ^{bcd}
23	38.16±0.060 ^{bcd}	37.39±0.728 ^{cdefg}	-0.92±0.035 ^{efg}	-0.92±0.038 ^{fghij}	11.64±0.040bc	10.76±0.806 ^{cde}
24	37.80±1.768 ^{bcde}	37.04±0.552 ^{defg}	-0.94±0.003de	-0.95±0.010 ^{bcd}	10.99±1.605 ^{bcde}	10.74±0.608 ^{cde}

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

Fatty Acid Composition. The fatty acid composition is an important quality parameter and authenticity indicator of virgin olive oils. The results are shown in Table 3 and a typical chromatogram was presented in Fig.2.

Filtration had no detectable effect on fatty acid composition. As expected oleic acid (C18:1) was the most abundant (68.64%) fatty acid followed by palmitic acid (C16:0) and linoleic acid (C18:1). Oleic acid (C18:1) contents of early harvest monocultivar olive oils produced in Turkey were between 62.41-80.26% [13,29-30]. Linoleic and linolenic acids, which are much more susceptible to oxidation than monounsaturated fatty acids (MUFA) were 12.09% and 0.7% respectively. Virgin olive oils are classified into two types based on their fatty acid compositions. Turkish, Spanish, Italian and Greek virgin olive oils characterized by low linoleic and palmitic, and high oleic acid contents are the first type, while Tunisian oils are the second type characterized by high linoleic and palmitic and low oleic acid contents [30]. The linolenic acid level of Turkish virgin olive oil samples was equal to the maximum value regulated by the Turkish Codex (Turkish Codex, 2010) and the EU (0.7%) (EEC, 2002).

Tocopherol Profile. Tocopherol (α , β , γ) profile of Uslu EVOO were determined every two months during storage (Table 4). The results showed that tocopherol

contents (α , β , γ) decreased with increasing storage time as expected. The lowest tocopherol contents were obtained after two year of storage. It means that 52% of α tocopherol, 15–20% of β -tocopherol and 10% of γ tocopherol contents were decomposed in both filtered and unfiltered samples during storage. Filtration had an important effect on tocopherols contents. The amounts of tocopherol isomers (α -tocopherol and γ -tocopherol) was higher in unfiltered samples. β -tocopherol content was higher in filtered samples. These results were in agreement with other researcher results [3-5,9,22].

Total Polyphenol. Total polyphenols contents of the samples were presented in Table 5. The highest total polyphenol values were determined at fresh oils and its amount decreased with time. But the decreases were not dramatic as well as tocopherols, after two years 55.63% and 49.63% of total polyphenols were decomposed in filtered and unfiltered samples, respectively. Filtered samples had higher total polyphenol content indicating filtration had a significant effect. It protect polyphenols from decomposition. After a short term or long term storage significant decreases in total polyphenols were reported for monocultivar and commercial olive oils by Clodoveo et al. [24]; Morelló et al. [8]; Abdalla et al. [23] and Baiano et al. [22].

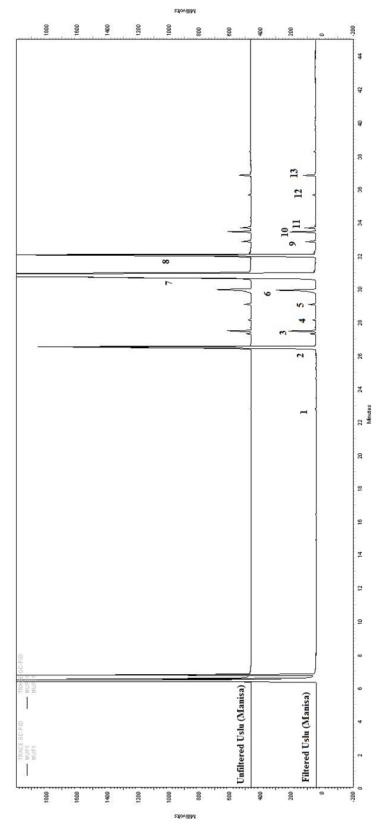


Fig. 2. Fatty acid profiles of extra virgin olive oils extracted from Uslu variety 1 14:0, 2 16:0, 3 16:1, 4 17:0, 5 17:1, 6 18:0, 7 18:1, 8 18:2, 9 18:3, 10 20:0, 11 20:1, 12 22:0, 13 24:0

Storage Period	a -Toco	opherol	β-Τος	opherol	y-Tocopherol	
(Month)	F	UF	F	UF	F	UF
0	237.28±3.112 ^a	239.66±0.212 ^a	1.37±0.006 ^a	1.21±0.009 ^a	0.84 ± 0.005^{a}	0.90±0.011ª
2	222.24±1.941 ^b	236.80±0.714 ^{ab}	1.23±0.008b	1.05±0.003b	0.68±0.014 ^b	0.76±0.004 ^b
4	214.96±2.398°	233.99±2.116 ^b	1.05±0.004°	0.84±0.003°	$0.48 \pm 0.008^{\circ}$	0.51±0.006°
6	198.73±0.233 ^d	223.45±2.842°	0.98±0.003 ^d	0.65±0.005 ^d	0.34±0.010 ^d	0.33±0.005 ^d
8	195.03±2.574 ^d	211.00±0.389 ^d	0.51±0.010 ^e	0.52±0.001e	0.22±0.004 ^e	0.22±0.007 ^e
10	170.38±4.043 ^e	156.77±1.121e	0.41 ± 0.006^{f}	0.48 ± 0.003^{f}	0.16 ± 0.005^{f}	0.18 ± 0.007^{f}
12	134.07±2.842 ^f	136.41±3.180 ^f	0.35±0.012 ^g	0.38±0.010 ^g	0.11±0.001g	0.12±0.003 ^g
15	130.33±0.065 ^{fg}	133.82±1.245 ^{fg}	0.30±0.011 ^h	0.34±0.009 ^h	0.11±0.005g	0.12±0.005 ^g
18	125.80±0.600gh	131.35±0.150g	0.25 ± 0.010^{i}	0.31 ± 0.002^{i}	0.10±0.005 ^{gh}	0.08 ± 0.005^{h}
24	123.31±0.043 ⁱ	124.34 ± 1.345^{h}	0.21±0.000 ^j	0.23 ± 0.012^{j}	0.09 ± 0.000^{h}	0.08 ± 0.000^{h}

Table 4 – Tocopherol content	of Uslu (Manis	a) monocultivar during	24 months' storage (ppm)

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

Table 5 – Changes in total phenols of EVOOs during24 months of storage (ppm)

Storage Period	Uslu	EVOO
(Month)	F	UF
0	449.56±0.940 ^a	407.64±4.051 ^a
1	353.83±0.417 ^b	388.54±1.980 ^b
2	350.71±0.800°	376.30±2.001°
3	344.19±0.445 ^d	364.29±1.655 ^d
4	340.62±0.572 ^e	355.34±4.766 ^e
5	337.06±2.043 ^f	346.99±0.000 ^f
6	332.91±0.594 ^g	344.56±0.255 ^f
7	330.79±0.120 ^h	336.15±2.977 ^g
8	325.02±1.088 ⁱ	322.44±1.803 ^h
9	323.86±0.268 ⁱ	311.25±1.414 ⁱ
10	321.22±0.035 ^j	302.41±1.187 ^j
11	316.74±1.180 ^k	294.06±2.560 ^k
12	308.83±1.8731	285.34±0.127 ¹
15	304.48±0.821 ^m	258.51±0.712 ^m
18	289.36±0.983 ⁿ	233.62±0.659 ⁿ
21	274.26±0.106°	216.13±0.158°
24	250.12±0.211 ^p	202.34±0.321 ^p

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

Phenolic Profiles. Phenolic profiles of Uslu (Manisa) EVOOs were determined during two years' storage both in filtered and unfiltered samples (Table 6-7). There are several papers on determination of Turkish olive oils in literature, this is the especial report related to effect of storage time on phenolic compounds of Uslu (Manisa) monocultivar extra virgin olive samples in both filtered and unfiltered types. 3-hydroxy tyrosol was identified in filtered Uslu (Manisa) sample second and sixth (6.43 and 6.93 ppm). 3,4-dihydroxy benzoic acid was detected in second month (4.13 ppm). Concentration of 3,4-dihydroxy benzoic acid was decreased in fourth and fifth months (0.17-0.18 ppm, respectively). This value increased sixth, seventh and eighth months (2.32, 6.57, 6.60 ppm respectively), it received to 11.60 ppm in eighteenth month. Final content of 3,4-dihydroxy benzoic acid was 9.56 ppm. Tyrosol was detected in second month (1.35 ppm). Amounts of tyrosol increased in fourth (1.74 ppm) and ninth months (3.47 ppm). Last detection of tyrosol was in tenth month (2.82 ppm). Initial concentration of 4-hydroxy benzoic acid was 0.98 ppm. The value of 4-hydroxy benzoic acid was altered during storage. The highest amount of 4-hydroxy benzoic acid was obtained in seventh month (1.70 ppm). The final concentration of 4-hydroxy benzoic acid was 1.01 ppm. 4-hydroxy phenyl acetic acid detected only in sixth month (2.74 ppm).

The highest amount of trans-ferulic acid had seen in fifth month (4.46 ppm). Value of trans ferulic in seventh and eighth months was 0.08 ppm. This value increased to 0.71 ppm at the end of storage. m-coumaric acid had 0.70 ppm in zeroth month. This value changed by time. The final concentration of m-coumaric acid was 0.38 ppm. o-coumaric acid was detected in first month with low concentration (0.05 ppm). The highest amount of o-coumaric acid was obtained in 2th month (0.60 ppm). This value decreased in third and fourth months. Final detection of o-coumaric acid was in sixth month (0.30 ppm). Oleuropein is an important phenolic compound that identified in first (1.31 ppm), second (1.15 ppm) and seventh (1.48 ppm) months.

Luteolin had the highest amount among phenolic compound that were identified in filtered Uslu (Manisa) sample. Amount of this polyphenol was 240.12 ppm in zeroth month and this value decreased in first and second months (235.77-226.5 ppm respectively). The value of luteolin was increased in next two months (293.47-312.57 ppm, respectively). Amount of luteolin was decreased from 212.97 to 93.57 ppm at the end of storage. Apigenin content of filtered Uslu (Manisa) was 2.38 ppm in zeroth month and increased to 3.11 ppm in third month. Amount of apigenin was decreased from 3.31(seventh month) ppm to 0.22 (twelfth month). It received to 2.85 ppm at end of two years' storage. Phenolic compounds of filtered Uslu (Manisa) is shown in Table 6.

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-86-0 0.0	1.35± 0.0	pu	1.74 ± 0.0	nd	pu	pu	pu	3.47± 0.0	2.82± 0.2	pu	pu	pu	pu
	pu .	0.0 ±20.1	pu	1.45±	1.48 ± 0.0	1.70± 0.1	1.5±0.0	pu	pu	1.43± 0.0	0'0 т99'1	1.24± 0.1	1.01± 0.0
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apigenin 2.38: 2.971 0.0 0.0	3.091	3.11 ±	3.01±0.0	$\frac{2.67}{0.0}$	2.631 0.0	3.31+ 0.0	3.36: 0.0	3.581 0.0	2.771 0.0	1.46+ 0.0	0.22+ 0.0	3.12+ 0.0	2.854 0.0

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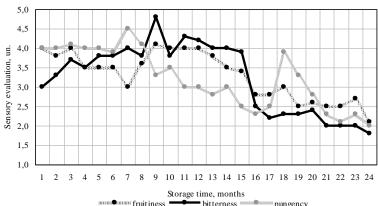
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		268.65±5.4	226.14+ 1.3	222.82: 2.9	285.62+ 4.3	325.56: 1.7	238.28+ 2.4	219.08+ 1.6	201.76+ 1.6	188.32+ 0.6	160.29+ 1.0	133.791 2.7	118.41: 2.8	105.18+ 2.4	102.01±0. 3	94.73±0.2
2.91±0.0 3.08±0.0 3.62±0.0 1.03±0.2 3.42±0.0 3.41±0.0 3.35±0.0 3.35±0.0	apigenin	2.91 ± 0.0	3.08±0.0	3.62±0.0	1.03 ± 0.2	3.45±0.0	3.41±0.0	3.35±0.0	3.33±0.0	3.33±0.2	3.23±0.3	1.0±70.5	2.93 ± 0.1	2.79 ± 0.0	2.61±0.0	2.13±0.0

Unfiltered Uslu (Manisa) EVOO had similar phenolic profile as filtered sample of Uslu. 3,4-dihydroxy benzoic acid was identified in first (2.67 ppm) and second month (3.55 ppm). This value decreased to 0.45 ppm in fourth month. Concentration of 3,4-dihydroxy benzoic acid was 3.67 and 7.95 ppm at sixth and seventh months. This value increased to 10.49 ppm at the end of two years storing. Tyrosol was detected in fourth month (1.63 ppm) and its contents were 3.02 and 2.94 ppm in ninth and tenth months, respectively. 4-hydroxy benzoic acid was 1.60 ppm in zeroth month, it was decreased to 1.00 ppm in third month. During fifth, sixth and seventh months, this values increased, but it was 1.62 and 1.83 ppm at eleventh and twelfth months. The final concentration of 4-hydroxy benzoic acid was 1.68 ppm. m-coumaric acid concentration was 0.97 ppm at zeroth month. Its content fluctuated during storage. The lowest content of m-coumaric acid was 0.30 ppm and its content was 0.39 ppm in tenth month. This value increased to 0.44 ppm at twenty-fourth month. ocoumaric acid was identified second month (0.07 ppm) and it was at 0.09 ppm after 24 months storing. Oleuropein was identified in fifth month (0.67 ppm) and its amount were 1.46,1.48, and 1.52 ppm in ninth, eleventh and twelfth months, respectively. Luteolin was detected as 268.65 ppm at the beginning and it was decreased to 222.82 ppm in second month. The maximum level of luteolin was 325.56 ppm in fourth month. It was decreased from sixth to twenty-fourth months and final amount of luteolin was 94.73 ppm. Apigenin concentration was 2.91 ppm in zeroth month and it increased to 3.62 ppm in second month. The minimum level of apigenin was 1.03 ppm in third month. This value increased to 3.45 ppm in fourth month. Between fourth to twelfth months, its content decreased to 2.79 ppm. Concentration of Apigenin was 2.13 ppm at the of 2 years storing. Phenolic compounds of unfiltered Uslu (Manisa) is shown in Table 7.

These results suggested filtration and storage caused to change phenolic profile confirmed by literature. It is widely recognized that the simple phenols, tyrosol and hydroxytyrosol, increase over time due to hydrolytic processes of the secoiridoidic derivatives representing their linked forms [2]. Yorulmaz et al [31] reported that luteolin was the most abundant phenolic compound following trans-cinnamic acid and luteolin-7-glucoside. They also quantified tyrosol, syringic acid, p-coumaric acid, luteolin-7-glucoside, trans cinnamic acid, luteolin and apigenin in Turkish olive oils extracted from different olive varieties. Montedoro and Servili [32] 3,4-DHPEA, p-HPEA, vanilic acid, cafeic acid, 3,4-DHPEA-EDA, 3,4-DHPEA-EA had been identified in olive oils. Morelló et al. [8] suggested that although storage did not appear to have any effect on vanilic acid or vanillin, which were present at low concentration there was a significant decrease in the concentration of the rest of the quantified phenolic compounds. That reduction was more marked in the secoiridoid derivatives such as 3,4-DHPEA-EDA, p-HPEA-EDA and 3,4-DHPEA-EA indicating a more active participation in the oxidative processes as they were more easily oxidized. Among the most representative phenolic compounds in olive oil, lignans seem to be the most stable during oil storage. Mulinacci et al. [2], and García et al. [33] showed an increase tyrosol and hydroxytyrosol contents over time due to hydrolytic processes of the secoiridoidic derivatives. Gómez-Alonso et al. [26] stated that the main phenols were the dialdehydic form of elenolic acid linked to tyrosol (p-HPEA-EDA), oleuropein aglycon, and the dialdehydic form of elenolic acid linked to hydroxytyrosol (3,4-DHPEA-EDA). Baiano et al. [22] reported that there were increasing and decreasing trends in phenolic compounds (3,4-DHPEA, p-HPEA, vanillin, pcoumaric acid, 3,4-DHPEA-AC, 3,4-DHPEA-EDA, p-HPEA-AC, p-HPEA-EDA, 1-acetoxipinoresinol + transcinnamic acid, p-HPEA-EA) content.

Sensory Analysis. Filtered EVOO of Uslu cvs was stable in pungency, fruitness and bitterness (Fig 3.). This event may be related to filtration process. The difference between 0 to 24 month is about 2 points for pungency and fruitiness but 1 point for bitterness. This variety has stabile olive oil so the flavor is protected for 24 months even in the room temperature.

In unfiltered Uslu EVOO, Pungency was higher than fruitness and bitterness (Fig 4.). The difference between 0 to 24 month is about 3 points for pungency and bitterness but 1 point for fruitiness. This variety has stable olive oil, so the flavor can be protected only 20 months even in the room temperature.



Filtered Uslu

Fig. 3. Sensory values of filtered Uslu (Manisa) olive oils during 24 months of storage time

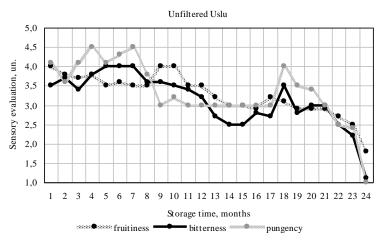


Fig. 4. Sensory values of unfiltered Uslu (Manisa) olive oils during 24 months of storage time

Conclusion

There is some limited research paper about Uslu EVOO in the literature, therefore this project can be useful for determination of its physical and chemical properties. On the hand, present study will be also useful for enrichment of Turkish olive oil database for better programming Republic of Turkey, Ministry of Food, Agriculture and Livestock. The effects of filtration process and storage time for 24 months on the chemical properties such as free acidity, peroxide value, color, UV absorbance, fatty acid composition, tocopherol content, total phenols, and phenolic compounds of monocultivar Extra Virgin Olive Oils (EVOOs) extracted from some Uslu (Manisa) produced in a mobile olive oil processing unit were investigated in present project. According to the results, Free fatty acid content of both filtered and unfiltered Uslu increased a little after 24 months of storage. This means that filtration had no effect on free fatty acid content of EVOOs. This trend was observed in peroxide value of Uslu (Manisa) EVOOs. Both filtered and unfiltered EVOOs' peroxide value increased during 24 months of storage. Filtration didn't affect peroxide value of EVOOs, EVOOs had stable trend during two years' storage time. Color of filtered and unfiltered Uslu (Manisa) altered from green to yellow during two years' storage. UV values of both filtered and unfiltered showed that Uslu EVOOs were in IOC standard for EVOO classification during storage time. Fatty acid profiles of filtered and unfiltered Uslu had no important alteration during three months of storing. Unfiltered Uslu (Manisa) had higher amount of α -tocopherol than filtered, but

degradation rate of a-tocopherol in both type was somehow similar together. Filtered Uslu (Manisa) had higher β -tocopherol than unfiltered, but y-tocopherol content unfiltered was more than filtered. It can be said that filtration had no very important effect on degradation of tocopherol isomers. Total polyphenol content of filtered Uslu (Manisa) was higher than unfiltered in initiation and at the end of the storage time. Luteolin was the most abundant phenolic compounds in filtered and unfiltered Uslu (Manisa) sample. It was verified other researcher results. Tyrosol content of both filtered and unfiltered Uslu (Manisa) had increase and decrease trend during 24 months of storage time. But hydroxyl tyrosol content increased in filtered Uslu during storage time. Oleuropein content of filtered and unfiltered Uslu was very close together, and its concentration increased during storage. According to chemical results, it can be said that total phenol, phenolic compounds, and tocopherol isomers of Uslu (Manisa) monocultivar EVOOs were decreased in both filtered and unfiltered. Uslu (Manisa) showed good oxidative stability during storage time because of high content of phenolic compounds and atocopherol. Present research, disclose some important and effective properties of Uslu EVOO extracted by Mobile Olive Oil Processing Unit for improving of Olive oil production and programming in Turkey.

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Хімія харчових продуктів і матеріалів / Chemistry of food products and materials

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