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Determination of Seasonal Changes in Olive Oil by Using Differential Scanning Calorimetry Heating Thermograms

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Abstract Thermal properties (upon heating) of extra virgin olive oils (EVOOs) from Aegean cultivars were determined to evaluate the possibility of using differential scanning calorimetry (DSC) as a tool for monitoring seasonal changes in chemical composition. Chemical properties of the samples were analyzed to assess the relationship between the thermal properties and chemical properties as well. The thermal properties of Aegean cultivars were found to be influenced by major components (palmitic acid, oleic acid, linoleic acid, triolein and palmitodiolein). The melting offset temperature, the temperature range of melting and the melting enthalpy of Aegean cultivars presented significant differences with respect to crop season ($p < 0.05$). DSC may be utilized to evaluate compositional changes with respect to crop season.

Keywords Chemical properties · Differential scanning calorimetry · Extra virgin olive oil · Thermal properties

Introduction

The increasing popularity of extra virgin olive oil has mainly been attributed to its unique sensory, nutritional, and biological properties. Recent studies have revealed that the high content of oleic acid and phenolic compounds in virgin olive oil contributes to the prevention of human disease [1, 2].

After the European Union, Turkey is one of the major producers of olive oil and accounts for 10% of total world exports. The Aegean region, the Marmara region, the Mediterranean region and the Southeast Anatolia region are olive growing regions in Turkey, which is the world's fifth largest producer of olive oil with an annual production of 120,000 tonnes. The Aegean coast, the major olive growing region, comprises 75–80% of the total national production. The Memecik cultivar, the dominant cultivar in the South Aegean region, makes up 45% of the total olive trees in Turkey, while the Ayvalik cultivar makes up 20% of the total olive trees and its growing area is located mainly in the North Aegean region [3, 4].

To conduct authentication studies regarding geographical origin and cultivar characterization, knowledge of the chemical characterization of olive oils is essential. In recent years, the chemical characterization of olive oil varieties has been studied by the main olive oil-producing countries [5]. These studies were carried out by evaluating the chemical parameters such as fatty acid, triacylglycerol and sterol compositions. However, these analytical techniques require time-consuming methods. Consequently, simple and quick methods are needed to characterize olive oil according to geographical origin and cultivar.

Differential scanning calorimetry (DSC) is a thermoanalytical technique used in oil research to determine the thermal properties of the oils which have been found to be affected by their chemical compositions. DSC application has been proposed as a tool for oil characterization [6–10]. Since the DSC method is quick and does not require sample preparation, it has some advantages over the classical characterization methods.

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The thermal properties of EVOO were found to be correlated with fatty acid and triacylglycerol compositions and the DSC application was proposed to discriminate monovarietal EVOOs according to cultivar [6, 7, 10]. DSC may be used to determine the changes in chemical composition related to crop season as well. However, none of the data in the literature had dealt with DSC application to evaluate seasonal changes of olive oil.

The chemical characterization of Aegean olive oils has been studied by some authors [11–13]. However, there is no available data elucidating the thermal properties of Aegean olive oils. The aim of this study was to determine the thermal properties (upon heating) of Aegean olive oils and to evaluate the possibility of using DSC as a tool for monitoring the changes in olive oil composition with respect to crop season.

Materials and Methods

Samples

Commercial monovarietal extra virgin olive oils were provided by TARIS (Olive and Olive Oil Agricultural Sales Cooperatives Union, Izmir, Turkey). Ayvalik oil samples (*n*:8) from the North Aegean region and Memecik oil samples (*n*:8) from the South Aegean region were collected during the 2006/2007 and 2007/2008 harvest seasons. Thirteen oils were extracted by using a triple-phase decanter and three oils with dual-phase decanter. The olives were harvested from October to December (black fruit) and immediately processed in the TARIS plant. Olive oil samples were stored in dark bottles at room temperature before analysis.

Solvents and Standards

All solvents used were analytical or LC (liquid chromatography) grade (Biosolve). Commercial standards of triacylglycerols and fatty methyl esters were purchased from Sigma–Aldrich.

Quality Parameters

Methods of free acidity and UV characteristics of Aegean olive oils can be found in Ilyasoglu et al. [3].

Fatty Acid Composition

The determination of fatty acid composition was carried out according to the analytical methods described in AOCS Official method Ce 1f-96. The oil samples were subjected to saponification with sodium hydroxide (0.5N) and methyl esterification with BF_3–methanol. The samples were injected into an Agilent Technologies 6890N gas chromatograph equipped with a flame ionization detector, a split/splitless injector and a long capillary column CP-Sil 88 (0.25 mm × 0.25 μm × 50 m, Varian-Chrompack). The column temperature was isothermal at 190 °C for 65 min and the injector and detector temperatures were both 220 °C. The carrier gas was hydrogen at a flow rate of 1 ml min^{-1}, the split ratio was 1:100, and the injection quantity was 1 μl. The identification of FAME was performed by using a standard FAME reference mixture. The peak areas were computed by the integration software and fatty acids were given in percentages relative to the total fatty acid contents.

Triacylglycerols (TAG)

Triacyglycerol analysis was carried out according to the method developed by Rombaut [14]. Oil samples were dissolved in a concentration of 0.5 mg ml^{-1} in dichloromethane/acetonitrile (30:70) prior to injection. The Thermo Finnigan HPLC system was equipped with four solvent lines, Alltima HPLC 18 HL, 3 μm, 150 × 3 mm column, autosampler, and Alltech ELSD 2000 Evaporative Light Scattering Detector. The detector temperature was set at 64 °C, and the flow rate of the mobile phase was 0.72 ml min^{-1}. Nitrogen was used as the nebulizing gas at a flow rate of 1.2 ml min^{-1}. The identification of TAG was performed by using TAG standards. The peak areas were computed by the integration software and the TAG results were given in percentages relative to the total triacylglycerol content.

Differential Scanning Calorimetry (DSC)

Oil samples (6–7 mg) were weighed in aluminum pans and analyzed with DSC Q10 (TA Instruments, New Castle, USA). The instrument was calibrated with a standard (indium and *n*-dodecane) and an empty pan was used as a reference. The purge gas, nitrogen, was delivered at 30 ml min^{-1}. The following temperature–time curve was applied: (a) oil samples were held at 50 °C for 3 min; (b) cooled to −40 °C at a rate of 5 °C min^{-1}; (c) kept isotherm at −40 °C for 6 min; (d) heated to 50 °C at a rate of 5 °C min^{-1}; (e) kept isotherm at 50 °C for 3 min. Universal Analysis Software (TA Instruments) was used to analyze the thermograms. Enthalpy change (ΔH), onset (T_{on}), and offset (T_{off}) temperatures and the temperature range of the transition (T_{range}) were obtained. The temperature range of the transition was calculated as temperature difference between T_{on} and T_{off}. To determine ΔH values of peaks, the linear option from integration menu was selected.

The DSC method used in this study was based on the methods developed by Ferrari et al. [15] and Tan and Che Man [16]. The parameters of the DSC method such as scanning rate, temperature and holding time were selected carefully to obtain reliable and reproducible results. Oil may be heterogeneous at room temperature that complicates to obtain reproducible thermograms. It was reported that sample homogenization was obtained by heating the oil to 50 °C for at least 3 min. The reproducibility of the DSC curves was improved by the thermal treatment as well [15]. Thus, 50 °C was chosen as the maximum temperature of the DSC application to provide homogenous samples. The minimum temperature of the DSC method was selected as −40 °C since good results were reported at this temperature [15, 17]. The studies on the use of DSC for EVOO characterization revealed that heating thermograms were more distinctive than cooling thermograms [10, 18]. Therefore, only the heating thermograms of Aegean olive oils were utilized to evaluate the calorimetric parameters of heat transitions.

Six independent samples were analyzed to calculate the repeatability of the DSC method. The standard deviation of the method was below 5% for each parameter.

Statistical Analysis

All experiments were carried out in triplicate. Two-way ANOVA was used to statistically evaluate differences according to cultivar and crop season. Pearson correlation and multiple regression analysis were performed to assess correlations between chemical compositions and thermal properties. Statistica 6.0 software (StatSoft Inc., Tulsa, OK, USA) was used to process the data.

Results and Discussion

Heating Thermogram

Oil samples were characterized by DSC heating thermograms and the thermal properties were obtained from the thermograms. The DSC representative heating thermograms for Ayvalik and Memecik cultivars are shown in Fig. 1 for the 2006/2007 and 2007/2008 crop seasons. All samples exhibited a minor exothermic peak (A), a major endothermic peak (B) at temperatures below ∼0 °C and a minor endothermic peak (C) at temperatures above ∼0 °C. In the literature, similar exothermic transition [10, 18] and endothermic transitions [19] were observed for olive oil and the exothermic event was reported to be associated with the rearrangement of TAG polymorphic crystals into a more stable form.

Triolein, the major TAG detected in all the samples, represented more than 50% of the total TAG (Table 1). The melting thermogram of pure triolein is shown in Fig. 2. A large endothermic peak centered at ∼5 °C was observed. A single endothermic peak centered at 3 °C was reported for pure triolein [20]. POO, another major TAG

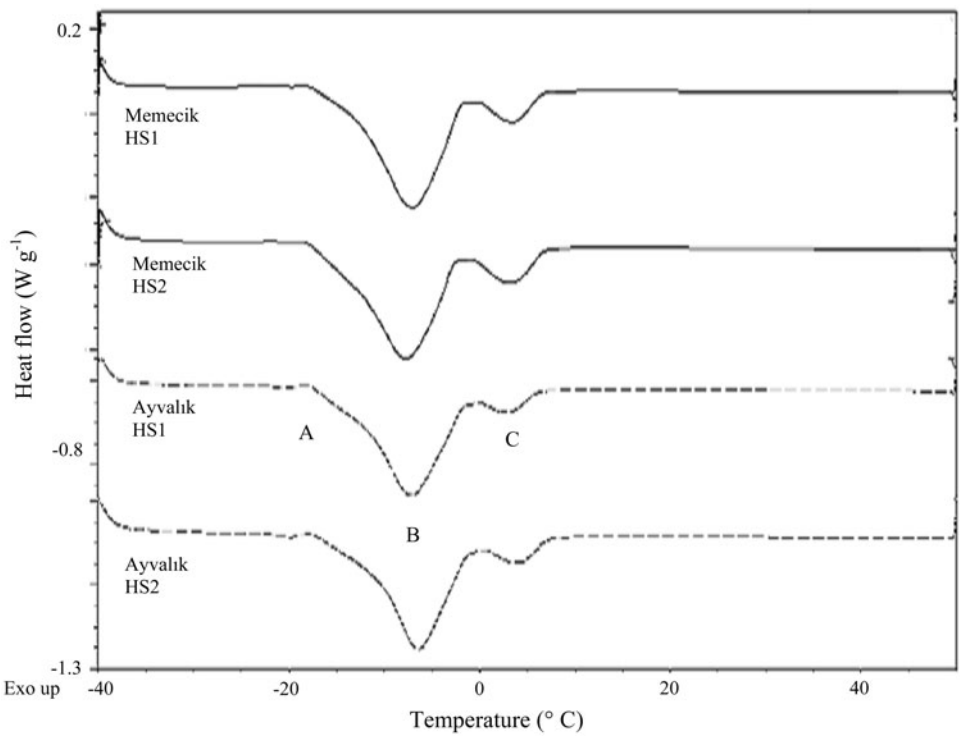

Fig. 1 Representative heating thermograms of Aegean olive oils HS1: 2006/2007 harvest season, HS2: 2007/2008 harvest season

Table 1 Means and standard deviations values for quality parameters, fatty acid and TAG compositions (expressed in %) of Aegean cultivars

Parameters	Ayvalik cultivar (mean ± SD) North Aegean		Memecik cultivar (mean ± SD) South Aegean	
	2006/2007 (*n*:3)	2007/2008 (*n*:5)	2006/2007 (*n*:4)	2007/2008 (*n*:4)
Quality parameters				
Free acidity (%)	0.49 ± 0.01	0.51 ± 0.01	0.64 ± 0.02	0.55 ± 0.02
K_{232}	2.45 ± 0.03	1.83 ± 0.05	2.26 ± 0.05	2.07 ± 0.07
K_{270}	0.14 ± 0.01	0.13 ± 0.01	0.15 ± 0.01	0.15 ± 0.01
Fatty acids (%)				
C16:0	13.01 ± 0.17	13.87 ± 0.36	12.64 ± 0.80	12.62 ± 0.40
C16:1	0.61 ± 0.02	0.70 ± 0.03	0.68 ± 0.06	0.67 ± 0.06
C18:0	2.83 ± 0.10	2.82 ± 0.06	2.51 ± 0.08	2.62 ± 0.18
C18:1	72.79 ± 0.36	71.08 ± 0.47	75.42 ± 0.38	75.17 ± 0.75
C18:2	10.06 ± 0.21	10.81 ± 0.17	7.98 ± 0.71	8.17 ± 0.24
C18:3	0.71 ± 0.02	0.68 ± 0.05	0.77 ± 0.03	0.76 ± 0.06
SFA	15.84 ± 0.17	16.70 ± 0.33	15.15 ± 0.73	15.24 ± 0.47
MUFA	73.40 ± 0.37	71.79 ± 0.46	76.10 ± 0.38	75.84 ± 0.70
PUFA	10.77 ± 0.20	11.49 ± 0.17	8.74 ± 0.72	8.92 ± 0.26
TAG (%)				
LOO	9.90 ± 0.28	10.38 ± 0.82	7.27 ± 1.38	7.46 ± 0.23
PLO	2.35 ± 0.13	2.92 ± 0.37	1.35 ± 0.10	1.73 ± 0.19
OOO	58.88 ± 2.25	56.79 ± 1.74	65.54 ± 2.24	64.11 ± 1.64
POO	24.02 ± 0.48	26.00 ± 1.10	21.85 ± 2.52	23.97 ± 1.27
POP	0.98 ± 0.23	1.06 ± 0.07	0.84 ± 0.19	0.91 ± 0.09
SOO	1.70 ± 0.06	1.91 ± 0.50	1.60 ± 0.39	1.31 ± 0.32
SUU	28.06 ± 0.60	30.82 ± 1.13	24.81 ± 2.70	27.00 ± 1.40
SSU	0.98 ± 0.23	1.06 ± 0.07	0.84 ± 0.19	0.91 ± 0.09
UUU	68.78 ± 1.99	67.17 ± 1.23	72.81 ± 3.59	71.57 ± 1.48

SFA saturated fatty acids, *MUFA* monounsaturated fatty acids, *PUFA* polyunsaturated fatty acids

SUU monosaturated TAG, *SSU* disaturated TAG, *UUU* triunsaturated TAG

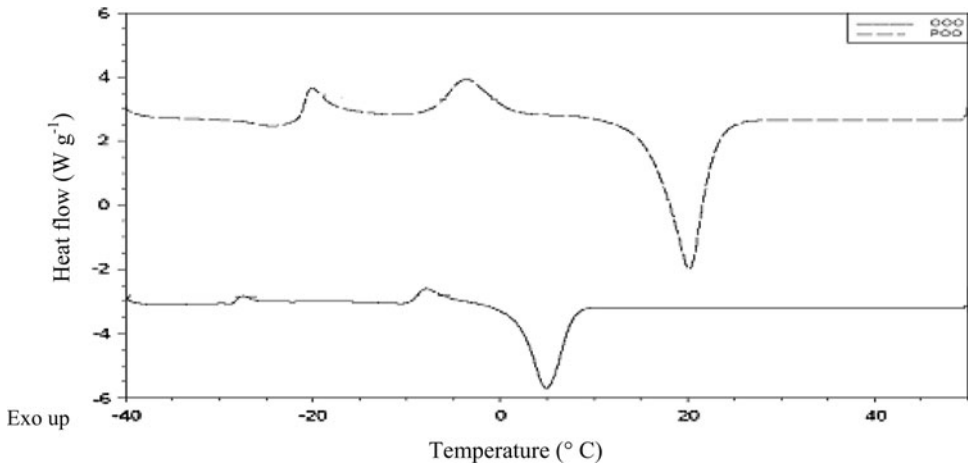

Fig. 2 Heating thermograms of triolein and palmitodiolein

detected in all the samples, showed a higher peak transition temperature than that of triolein (Fig. 2). The data found in the literature [10, 18–20] and our results indicated that the large endothermic peak was related to the melting of triunsaturated TAG, especially triolein and the minor peak was related to the melting of monosaturated TAG, especially POO.

The exothermic and endothermic transitions exhibited different peak profiles among the studied cultivars. The exothermic peak was more evident in Ayvalik oils. Furthermore,

Table 2 Thermal properties of Aegean cultivars

Thermal properties	Ayvalik cultivar North Aegean		Memecik cultivar South Aegean	
	2006/2007 (*n*:3)	2007/2008 (*n*:5)	2006/2007 (*n*:4)	2007/2008 (*n*:4)
T_{on} (°C)	−22.30 ± 0.11	−22.25 ± 0.45	−20.99 ± 0.78	−21.05 ± 0.47
T_{off} (°C)	6.31 ± 0.16	7.19 ± 0.49	6.58 ± 1.25	7.47 ± 0.32
T_{range} (°C)	28.61 ± 0.23	29.44 ± 0.71	27.58 ± 0.59	28.52 ± 0.41
ΔH (J g^{-1})	54.23 ± 0.84	51.11 ± 1.91	60.28 ± 6.38	55.26 ± 2.72

the shoulder at the beginning of the major endotherm was more distinguishable for Ayvalik oils. However, the minor endotherm was smaller in Ayvalik oils. Chemical properties of Aegean olive oils are presented in Table 1. With the exception of palmitic acid, the fatty acid composition showed significant differences among the studied cultivars ($p < 0.01$). The TAG composition presented significant differences between Ayvalik and Memecik cultivars ($p < 0.01$), except POP and SOO. The differences observed between the peak profiles of Ayvalik and Memecik oils may be related to variations in the chemical composition. Thermal properties of EVOO were found to be influenced by major components (fatty acids and TAG) [7, 10, 18]. It was reported that minor components affected thermal properties of EVOO as well [10]. The minor components interfere with the major components (e.g. TAG), which may hinder the rearrangement of TAG into more stable polymorphic forms [8]. The adsorption of minor components into the crystal lattice of TAG forms mixed crystals, which melt at lower temperature than pure TAG crystals that have polymorphic forms exhibiting different melting temperatures. OOO was reported to have four polymorphic forms melting at −12, −8, −5 and 5 °C, respectively [10]. However, scarce information is available on the polymorphic behavior of TAG in olive oil. Minor components with TAG constitute a heterogeneous structure, which may promote polymorphic transitions.

Influence of Crop Season on Thermal Properties

Heating thermal properties were obtained from the onset of the exothermic/endothermic event to the offset of the endothermic event. The thermal properties (T_{on}, T_{off}, T_{range} and ΔH) of each cultivar are presented in Table 2.

A Pearson correlation analysis was performed to determine the correlation between the thermal properties and the chemical properties of Aegean olive oils. Correlations were found between the thermal properties and chemical properties. Pearson correlation coefficients are presented in Table 3. The melting onset temperature (T_{on}) was found to be positively correlated with palmitoleic acid ($r = 0.54$), oleic acid ($r = 0.74$), linolenic acid ($r = 0.70$), and triolein ($r = 0.66$), whereas it was found to be negatively correlated with stearic acid ($r = -0.88$), linoleic acid ($r = -0.86$), LOO ($r = -0.93$), and PLO ($r = -0.79$). A positive correlation was observed between the melting offset temperature (T_{off}) and palmitic acid ($r = 0.78$), palmitoleic acid ($r = 0.75$) and POO ($r = 0.69$). The temperature range of melting (T_{range}) was found to be positively correlated with palmitic acid ($r = 0.89$), stearic acid ($r = 0.60$), linoleic acid ($r = 0.67$), LOO ($r = 0.62$), PLO ($r = 0.85$), POO ($r = 0.90$), and POP ($r = 0.60$). On the contrary, a negative correlation was found between T_{range} and oleic acid ($r = -0.85$), linolenic acid ($r = -0.53$), and OOO ($r = -0.84$). A negative correlation was observed between the enthalpy changes (ΔH) of Aegean olive oils and the thermal properties such as palmitic acid ($r = -0.91$), linoleic acid ($r = -0.54$), PLO ($r = -0.69$), POO ($r = -0.92$), POP ($r = -0.73$), whereas the values of ΔH were found to be positively correlated with oleic acid ($r = 0.76$) and OOO ($r = 0.78$). The results obtained in this study indicated that thermal properties (upon heating) of EVOO were largely influenced by the content of major components such as palmitic acid as SFA, oleic acid as MUFA, linoleic acid as PUFA, OOO as triunsaturated TAG, and POO as monosaturated TAG.

The T_{off} ($p < 0.05$), T_{range} ($p < 0.01$) and ΔH ($p < 0.05$) values of Aegean olive oil samples showed significant differences with respect to crop season. The T_{off} values of the samples from the 2007/2008 harvest season were found to be higher than those of the samples from the 2006/2007 harvest season (Table 2), which may be explained by the positive correlation (Table 3) observed between T_{off} and both palmitic acid and POO. Higher values of the T_{range} were obtained for the samples belonging to the 2007/2008 crop season, which may be related to the positive correlation (Table 3) found between T_{range} and palmitic acid, linoleic acid and POO and the negative correlation between T_{range} and both oleic acid and triolein. The samples belonging to the 2006/2007 harvest season presented the highest values of ΔH compared to the 2007/2008 harvest season. These differences may be attributed to the positive correlation between ΔH and both oleic acid and triolein, as well as the negative correlation between ΔH and palmitic acid, linoleic acid and POO. Our results could not be compared to the literature since there is no such data to be found there.

Table 3 Pearson correlation coefficient between thermal properties and chemical properties

Thermal properties	Chemical characteristics		
	Acidity	K_{232}	K_{270}
T_{on}	0.166	−0.018	0.192
T_{off}	−0.238	−0.439	−0.151
T_{range}	−0.366	−0.377	−0.311
ΔH	0.202	0.288	0.056

	Chemical characteristics								
	C16:0	C16:1	C18:0	C18:1	C18:2	C18:3	SFA	MUFA	PUFA
T_{on}	−0.204	**0.536**	−0.883	**0.740**	−0.864	**0.699**	−0.393	**0.757**	−0.858
T_{off}	**0.781**	**0.751**	−0.234	−0.188	−0.137	0.125	**0.645**	−0.161	−0.135
T_{range}	**0.888**	0.182	**0.601**	−0.847	**0.670**	−0.529	**0.939**	−0.839	**0.666**
ΔH	−0.909	−0.286	−0.456	**0.762**	−0.541	0.379	−0.924	**0.750**	−0.540

	Chemical characteristics								
	LOO	PLO	OOO	POO	POP	SOO	MSTAG	DSTAG	TUTAG
T_{on}	−0.933	−0.788	**0.657**	−0.300	−0.279	−0.055	−0.428	−0.279	0.392
T_{off}	−0.263	0.144	−0.267	**0.696**	0.386	0.019	**0.564**	0.386	−0.504
T_{range}	**0.620**	**0.853**	−0.841	**0.899**	**0.602**	0.068	**0.898**	**0.602**	−0.811
ΔH	−0.394	−0.689	**0.784**	−0.917	−0.725	−0.294	−0.905	−0.725	**0.856**

Marked correlations are significant at $p < 0.05$

Conclusion

The results of our study indicated that thermal properties varied from one crop season to the next. Thermal properties (upon heating) of EVOOs were found to be correlated with palmitic acid, oleic acid, linoleic acid, triolein and palmitodiolein content. Furthermore, seasonal changes in thermal properties were mainly influenced by palmitic acid, oleic acid, linoleic acid, triolein and palmitodiolein content. Consequently, it may be interpreted that DSC heating thermograms with their characteristic peaks may be utilized to follow the changes in the chemical composition related to the seasonal changes.

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