Olive Oil Using Gas Chromatography

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Olive oil is among the most popular supplements of the Mediterranean diet due to its high nutritional value. Therefore, the authenticity of extra virgin olive oil (EVOO) and virgin olive oil (VOO) is of major importance for both financial and health-related reasons. However, at the same time, because of economical purposes, it is also one of the products most subjected to adulteration. As a result, authenticity is an important issue of concern among authorities and consumers. Especially, regarding consumers, they require high-quality food products which a real characterized botanical and geographical origin. To this end many analytical techniques able to identify geographical and botanical origin and consequently, guarantee its quality and authenticity, have been developed.

Keywords: olive oil ; authentication ; adulteration ; volatiles ; gas chromatography ; solid phase micro extraction

1. Introduction

According to the International Olive Council (IOC), olive oil can be defined as the product of the fruits from *Olea europea* L. trees, obtained physically or mechanically by washing, decantation, centrifugation and filtration. These processes alter neither the nutritional composition of the final product nor interfere with its organoleptic characteristics, namely, taste and aroma. Regulation of the European Union (Reg. (EEC) 2568/91) and IOC have introduced specific requirements, according to which olive oil is classified in different categories which (a) define its quality, namely: alkyl esters of fatty acids composition (which are constituents connected to quality decline when their estimated value is greater than that those imposed by legislative authorities), degree of acidity (expressed as a percentage of g of oleic acid in 100 g of oil), determination of peroxides (expressed as milliequivalents of active oxygen per kilogram which oxidize potassium iodide), spectrophotometric indices and sensory characteristics (flavor and aroma) and (b) are characteristic parameters of its purity such as quantification of waxes (the limit is set at 150 mg/kg for EVOO and VOO), sterols (total sterol content (mg/kg) as well as % of free sterols), 2-glyceryl mono-palmitate [1]. This classification includes Extra Virgin Olive Oil (EVOO), which is directly produced from olives using mechanical means. EVOOs' aroma and taste are attributed to the presence of volatile and non-volatile compounds and their free acidity is lower than 0.8%. Virgin Olive Oil (VOO) is produced similarly to EVOO; however, its free acidity is higher but not greater than 2%, and its organoleptic properties are of inferior quality with respect to EVOO. Olive Oil (OO), is a mixture of refined and edible virgin olive oil, with acidity lower than 1%, and it is often labeled as virgin, light or pure olive oil. Refined olive oil is obtained from lower quality VOOs in terms of the degree of acidity and organoleptic characteristics. However, during refining processes such as deacidification, deodorization and decolorization, which require the use of organic solvents, these "defects" are usually eliminated. In particular, deodorization aims at removing off-flavor compounds from VOO, among which volatile compounds are included, that are produced via degradation/oxidation pathways due to long/inappropriate storage of olives before processing and/or of VOO. Deodorization requires high temperature under vacuum, which gradually degrades those unpleasant volatile compounds. Ordinary virgin oil is produced from olive oil fruits using the same processes as for EVOO; however, its acidity is 3.3%. This category has been excluded by the EU classification of olive oil, a strategy that will also follow other regulatory organizations. Lampante virgin olive oil is produced from low-quality fruits and is not destined for consumption. Its level of acidity is higher than 2%, and its organoleptic characteristics are rather poor (displeasing taste and smell). Finally, a distinct category that includes three subcategories is that kind of oil derived from pomace. Pomace is the residue of olive oil fruits after mechanical extraction of EVOO, which is treated with organic solvents, most common of which is hexane. Pomace oils include crude olive pomace oil, which is not adequate for human consumption; refined olive pomace oil, which is also not proposed for consumption and olive pomace oil, derived from olive pomace and VOO and is considered ideal for culinary purposes.

EVOO is a mixture of numerous bioactive compounds, the majority of which belong to monounsaturated fatty acids (MUFAs) such as oleic acid (18:1, n-9) and palmitoleic acid (16:1, n-7), with the first one being presented in abundance, and polyunsaturated fatty acids (PUFAs) such as linoleic acid (18:2, n-6) and linolenic acid (18:3, n-3). Other compounds such as vitamins (tocopherols), pigments such as β-carotene and lutein, phenolic compounds such as secoiridoids

(derivatives based on hydroxytyrosol and tyrosol structures) and flavones (apigenin, luteolin) and hydrocarbons such as squalene, a terpenoid hydrocarbon and precursor of the biosynthesis of steroids, are found in minor quantity; however, they are endowed with important biologic activity [2][3]. Consequently, due to its rich composition in nutrients of unique nutritional value, many health claims have been made for its health-promoting properties [4][5][6]. Hence, a steady and rapid increase regarding its demand, inside and outside the Mediterranean basin, is observed. However, the high nutritional value, in combination with high production costs and high demand, makes olive oil a targeted product fraud. Therefore, economically motivated adulteration incidents and cases of fraudulence are continuously revealed.

In order to avoid any kind of adulteration, the olive oil market is closely monitored by various organizations, which have suggested criteria and limits that should be respected by producers and distributors. Quality criteria including chemical composition parameters, organoleptic characteristics (aroma, taste, flavor), and guidelines of analysis methods have been proposed by the IOC and the Codex Alimentarius (CODEX STAN 33-1981). These proposed standards are not mandatory; however, they should be respected by the countries that agree to follow them. On the other hand, regulation of the European Community (EEC) No 2568/91 imposes compulsory parameters regarding quality and purity criteria, that should be adopted by the EU members. Furthermore, with the European Union regulation No 29/2012, geographic-origin labeling becomes a prerequisite.

Various analytical techniques, able to detect possible adulteration and consequently ensure its quality and authenticity, have been developed. Among them, gas chromatography is the most used technique regarding authentication issues that deal with geographical and botanical discrimination.

2. Detection of EVOO and VOO adulteration by Gas Chromatography

A common technique used by many researchers is solid-phase microextraction (SPME) accompanied with gas chromatography, which allows the characterization and quantification of the volatile composition of olive oil. SPME is a routine, low-cost, easy and efficient technique developed by Arthur and Pawlszyn in 1990 [7]. The technique occupies the use of a fiber coated with an extracting phase which can be solid or liquid, able to extract volatile and semi-volatile compounds from a sample. The sample is exposed to the fiber under steady temperature and stirred for a certain time, until the extracted compounds are absorbed by the fiber to the maximum of their concentration. Then, the SPME fiber can be directly injected into the GC system, where helium carries the volatile compounds into the capillary column and analysis of the sample begins.

Flores et al. (2006) [8] studied the adulteration of EVOO with hazelnut oil using various techniques based on its volatile fraction, among which SPME-multidimensional gas chromatography (SPME-MDGC). For the experiment, a Divinylbenzene/Polydimethylsiloxane (PDMS/DVB) fiber was used. Authors preferred to label an EVOO as adulterated only when both R and S enantiomers of filbertone were identified after an analysis of the results. Detection of adulteration was achieved to a range of 7–25% for hazelnut oil. One of the most important findings of this study was the fact that the hazelnut oil used was derived from unroasted raw material. This is important because levels of filbertone in this type of oil are lower than those derived from roasted hazelnuts oils [9]. Mildner-Szkudlarz et al. (2008) [10], using SPME/gas chromatography combined with mass spectrometry (GC-MS) and PCA, showed that gas chromatography could detect adulteration of an EVOO with hazelnut oil at 5% (v/v). Chemically, the volatile profile of these two oils is different. For example, EVOO is characterized by a high level of aldehydes (68%), whereas in olive oil of minor quality, the respective percentage is lower (38%), while ketones and acids constitute the majority. In this study, compounds that belong to the aldehydes family, as well as other compounds, helped distinguish EVOO from hazelnut oil, and these include E-2-hexenal, hexanal, pentanal, acetic acid, 2-octanone and 2-heptanone. Furthermore, many compounds such as propanoic acid, toluene and butanoic acid are present only in hazelnut oil. Consequently, based on quantitative and qualitative differences of the chemical profile between EVOO and hazelnut oil and by using chemometrics, adulteration can be detected.

2.1. Volatile Profile of EVOO and VOO as an Index for Evaluating Its Authenticity by Gas Chromatography Based on Geographical Origin

Olive oil volatile profile was studied by Cajka et al. (2004) [11], who aimed at distinguishing between EVOOs of different geographical origins. They used headspace (HS)-SPME followed by gas chromatography-ion trap mass spectrometry (GC-ITMS) combined with PCA, LDA and an artificial neural network with multilayer perception (ANN-MLP). The authors tested various types of fibers for their efficacy; however, they concluded that the most suitable was the DVB/CAR/PDMS (50/30 μm) fiber. Samples were analyzed with the GC-ITMS technique, yet authors proposed that for those compounds where GC chromatographs were not clear, Two-Dimensional Gas Chromatography and Time-of-Flight Mass Spectrometry (GCXGC-TOFMS) was the adequate analysis to perform. Indeed, spectra quality was significantly improved. Regarding multivariate methods, hallmark compounds such as alcohols, aldehydes and esters were chosen in order to build the chemometric model. A PCA analysis was first performed, and afterward, the LDA analysis followed. Nevertheless, the last

analysis was rather poor in classifying samples. In this regard, ANN-MLP analysis was performed, which definitely predicted an acceptable classification value. In another study performed by García-González et al. (2010) [12], monovarietal VOOs, produced in different regions of Chile, were distinguished based on their volatile and phenolic composition. Regarding volatile compounds, they were collected using the SPME method and a DVB/CAR/PDMS 50/30 μm fiber. Volatiles were separated with GC. Data were then handled with PCA and Analysis of Variance (ANOVA), and successful classification of the samples showed that climatic, cultivar and soil conditions are parameters that allow differentiation between monovarietal olive oil and determination of their geographical origin. In addition, the authors compared two varieties produced in Chile with the respective varieties produced in Spain. They concluded again that the volatile profile based on quantitation calculations is a marker that helps in distinguishing the country of provenance. Berlioz et al. (2006)[13] analyzed French virgin olive oils with Protective Designation of Origin (PDO) in order to confirm their authenticity by comparing their volatile profile with commercial EVOO. Volatiles were absorbed on a Divinylbenzene/Carboxen/Polydimethylsiloxane (DVB/CAR/PDMS) 50/30 μm fiber and then separated with the GC-FID technique. Results were first subjected to PCA analysis, where significant differences were observed between the PDO oils and the control ones. In particular, decreased amount of E-2-hexenal at the commercial EVOO was a key result that allowed discrimination between PDO and commercial oils. Finally, SIMCA analysis gave a good classification model as constructed by data based on particular compounds, characterized as marker compounds. A similar study was performed by Pizarro et al. (2011) [14], who related the geographical origin of EVOOs from various Spanish regions with the volatile profile. HS-SPME/GC-MS technique was performed and chemometric analysis, namely, PCA and Stepwise Linear Discriminant Analysis (SLDA), were used to explain results and classify the studied samples. Classification of the samples with the PCA analysis was not satisfactory; however, preliminary information regarding the formation of the classification model was obtained. On the other hand, six characteristic compounds were chosen in order to perform the SLDA analysis, and results showed a 100% discrimination rate among the different varieties. In another study, EVOOs from different countries were classified according to their geographical origin using the sesquiterpenes profile as a marker. Samples were analyzed with the HS-SPME-GC-MS technique with a DVB/CAR/PDMS 50/30 μm fiber, and PCA analysis was performed. The classification model was constructed with the PLS-DA analysis, and results were analyzed using both profiling and fingerprinting analytical approaches. The non-targeted approach gave better classification rates. In particular, all samples were 100% correctly classified, whereas the targeted approach varied from 46 to 100% [15]. Likewise, geographic and variety differentiation of VOOs was studied regarding Italian VOOs. SPME, GC-MS and gas chromatography with flame ionization detection (GC-FID) techniques were used to collect, identify and quantify, respectively, the results. Samples were characterized by the presence of C6 and C5 compounds, while other compounds such as *cis*-copaene, *cis*-farnesene presented significant quantitative differences between the varieties. Differences regarding the cultivars were studied with ANOVA analysis while DA combined with PCA was used for classification, based on their geographic region. A 100% correct classification rate was achieved [16]. Youssef et al. (2011) [17] also studied the influence of the geographical region on the aroma of VOOs from the same variety. The authors used HS-SPME, GC-MS and GC-FID methods to evaluate the chemical profile of the samples. Results showed that samples did not present significant differences regarding their qualitative profile; however, quantitative differences exist and helped differentiate VOOs according to their geographical origin with ANOVA, PCA and Hierarchical Cluster Analysis (HCA).

2.2. Volatile Profile of EVOO and VOO as an Index for Evaluating Its Authenticity by Gas Chromatography Based on Variety/Botanical Origin

Kaftan and Elmaci (2011) [18] studied the differences of the volatile compounds between two VOO varieties from Turkey (Ayvalik and Memecik) using the SPME/GC/MS technique. The fiber used was the PDMS/DVB/CAR 50/30 μm. Cluster and PCA analyses were performed to model results. Authors found that between these two varieties, qualitative and quantitative differences based on the presence of characteristic volatile compounds can lead to their differentiation. In particular, for the Ayvalik variety, hexanal, 3-hexen-1-ol, *cis*-3-hexenol and 9-octadecenoic acid were the discriminating compounds, whereas, for Memecik variety, 2-hexanal and 3-hexen-1-ol acetate were the most characteristic compounds.

Kosma et al. (2016) [19] studied the volatile and fatty acid profile of six cultivars of EVOO produced in Greece. The volatile profile was evaluated with the HS-SPME-GC/MS method using a DVB/CAR/PBMS 30/50 μm fiber. Compounds that belong to the C6 including (*E*)-3-hexen-1-ol, (*E*)-2-hexen-1-ol, 1-hexenol, (*Z*)-3-hexenal, hexanal, (*E*)-2-hexenal, (*E, E*)-2, 4-hexadienal and hexane. (*E*)-2-hexenal and C5, namely, 1-penten-3-ol, 1-pentanol, cis-2-pentenol, 1-penten-3-ne, 2-pentanone, 3-pentanone, pentanal, (*E*)-2-pentenal, pentane, (*Z*)-1,3-pentadiene and 3-methyl butanal group, as well as esters, hydrocarbons and terpenes were identified. According to multivariate analysis of variance (MANOVA), 34 samples were chosen to perform the LDA analysis. Differentiation between cultivars was successfully achieved and presented a high classification rate which corresponds to 83%. Croatian VOOs were studied by Lukic et al. (2019) [20], who attempted to differentiate them according to their geographical and variety origin. HS-SPME technique was used to collect volatile compounds with the use of a DVB/CAR/PBMS fiber. Conventional mono-dimensional GC-MS in combination with GC-

TOF-MS were used to separate the collected volatiles. Since TOF-MS is a technique known for its better selectivity and accuracy, more compounds that cannot be detected with the simple GC-MS were identified. In combination with chemometric analysis (ANOVA, PCA and SLDA), samples were successfully differentiated, whereas the presence of monoterpenes, sesquiterpenes and terpenes was determinant for their classification.

The volatile profile of six Algerian VOO varieties was studied by Nigri et al. (2012) [21]. HS-SPME (DVB/CAR/PBMS 50/30 μm fiber) followed by GC-MS was used to identify compounds. No chemometrics analysis was performed, and characterization of each variety was based on quantitative differences among the tested samples, which permitted their differentiation according to geographic criteria.

Mono and sesquiterpenes profiles of VOOs from different varieties derived from Spain and Italy were studied in order to determine differences that could lead to their differentiation [22]. The authors examined several parameters regarding the HS-SPME method, including the type of the fiber, extraction and temperature time, to find the best conditions which give the optimum chromatographic profile as derived from the GC-MS technique. They concluded that a DVB/CAR/PDMS fiber and the extraction temperature significantly affected the results obtained, on the contrary to the extraction time, which played no particular role. Quantitative differences of the studied compounds were more evident regarding sesquiterpenes, even though minor differences of the monoterpenes profile were also detected between some varieties. Taking into account only the mentioned quantitative differences, the authors supported that mono and sesquiterpenes can be a reliable index in distinguishing virgin olive oils based on cultivar and geographical parameters. In the study of Luna et al. (2005) [23], VOOs varieties from different countries but grown exactly under the same conditions (climate, irrigation, region) were examined in order to determine differences that could lead to their geographic identification. Dynamic headspace gas chromatography was used, and statistical analysis, including Brown–Forsythe test and SLDA was followed. Based on a strict number of compounds as derived from the Brown–Forsythe test, Spanish from Greek and Greek from Italian varieties were 100% correctly classified while between Spanish and Italian samples, the correct classification rate was 100% and 71.4%, respectively. Blasi et al. (2019) [24] classified EVOOs of different varieties according to their FA and volatile composition. In particular, regarding the volatile fraction, SPME/GC-MS method was used. Qualification and quantification of the compounds detected revealed that E-2-hexenal was the most abundant compound presented in all the studied varieties, followed by trans-2-hexen-1-ol. Based on the quantitative differences of these two compounds, successful classification of the samples according to their variety was achieved using the LDA analysis.

Pouliarekou et al. (2011) [25] examined VOOs from Western Greece. The SPME (DVB/CAR/PBMS 30/50 μm fiber) and GC/MS techniques were used to collect and separate volatile compounds. As mentioned above, in this study, most of the identified compounds belonged to the C6 and C5 family. ANOVA and LDA analysis were chosen for the classification of the results according to cultivar and geographical factors, and 74 and 87.2% correct classification rate, respectively, was achieved. Discrimination of the three most commercial Greek olive oil cultivars has also been achieved by SPME GC/MS in combination with the stepwise LDA and QDA algorithms. The correct classification rate reached 97.4 and 100%, respectively. The major volatile compounds selected for the discrimination of samples were terpenoid hydrocarbons [26].

3. Current Insights

According to ISO 9000, quality is defined as “the degree to which a set of inherent characteristics of an object fulfills requirements”. These requirements are imposed via rules and regulations by official authorities in order to combat fraud problems. Regarding olive oil, and especially EVOO and to a lesser extend VOO, issues of authentication are a continuous concern for authorities. Authentication issue includes a check of possible adulteration of EVOO with other types of oil of lower value and minor quality, misleading geographical indication and deceptive botanical origin.

EVOO authenticity is guaranteed by its unique, peculiar aroma owned to the presence of multiple volatile compounds that belong to different chemical groups, including aldehydes, ketones, alcohols and esters [27][28][29]. Specifically, the presence of C6 and C5 aldehydes and alcohols as well as their esters, are characteristic compounds to which the sensory quality of EVOO is attributed. These compounds are produced by the lipoxygenase (LOX) pathway and are presented at high concentrations in EVOOs, on the contrary, lower quality oils are present in minor quantities or traces or are even absent [30]. Although the volatile composition of an olive oil strongly depends on climatic, geographical, cultivar and extraction methods, various analytical techniques have been developed, able to characterize the oil’s authenticity, variety and geographical origin based on the volatile fraction. Differentiation of EVOOs and VOOs is a project studied by many researchers, who have concluded that, among others, certain molecules such as monoterpenes, sesquiterpenes and terpenes are characteristic compounds that allow the effective classification of the studied samples [19][20][22]. The volatile profile of an olive oil, as defined by SPME combined with the gas chromatography technique, has been proven to be a valuable tool to determine authenticity.

Literature data demonstrate that gas chromatography is a popular method to use when geographic origin should be declared. Olive oil is a complex mixture; however, as demonstrated by many studies, chemical interpretation of this mixture is achievable through gas chromatography. Consequently, a vast amount of data that describe the chromatographic profile of the examined olive oil are available, which, in combination with chemometric methods that point out differences between the studied samples, provide valuable information. In this regard, gas chromatography is a suitable method to use when the geographic origin of olive oil is required.

References

1. The European Commission. Commission Regulation (EEC) No. 2568/91 of 11 July 1991 on the Characteristics of Olive Oil and Olive. In Residue Oil and on the Relevant Methods of Analysis; The European Commission: Brussels, Belgium, 1991; pp. 1–83.
2. Blekas, G.; Tsimidou, M.; Boskou, D.. Olive Oil Composition, 2nd ed.; AOCS Press: Urbana, IL, USA, 2006; pp. 23.
3. Aparicio, R.; Harwood, J.. Handbook of olive oil: Analysis and properties. 2nd ed.; Springer: New York, NY, USA, 2013; pp. 677-714.
4. Owen, R.W.; Giacosa, A.; Hull, W.E.; Haubner, R.; Würtele, G.; Spiegelhalder, B.; Bartsch, H.; Olive-oil consumption and health: The possible role of antioxidants.. *Lancet Oncol.* **2000**, *1*, 107-112, .
5. Lozano-Castellón, J.; López-Yerena, A.; Rinaldi de Alvarenga, J.F.; Romero Del Castillo-Alba, J.; Vallverdú-Queralt, A.; Escribano-Ferrer, E.; Lamuela-Raventós, R.M.; Health-promoting properties of oleocanthal and oleacein: Two secoiridoids from extra-virgin olive oil. . *Crit. Rev. Food Sci. Nutr.* **2020**, *60*, 2532–2548, .
6. Lockyer, S.; Rowland, I.. Authorised EU health claims for polyphenols in olive oil In Woodhead Publishing Series in Food Science, Technology and Nutrition, Foods, Nutrients and Food Ingredients with Authorised EU Health Claims; Woodhead Publishing: Shaxton, UK, 2014; pp. 212-228.
7. Catherine L. Arthur; Janusz. Pawliszyn; Solid phase microextraction with thermal desorption using fused silica optical fibers. *Analytical Chemistry* **1990**, *62*, 2145-2148, 10.1021/ac00218a019.
8. Gema Flores; María Luisa Ruiz Del Castillo; Gracia Patricia Blanch; Marta Herráiz; Detection of the adulteration of olive oils by solid phase microextraction and multidimensional gas chromatography. *Food Chemistry* **2006**, *97*, 336-342, 10.1016/j.foodchem.2005.04.021.
9. M. L. Ruiz Del Castillo; G. Flores; M. Herraiz; G. P. Blanch; Solid-Phase Microextraction for Studies on the Enantiomeric Composition of Filbertone in Hazelnut Oils. *Journal of Agricultural and Food Chemistry* **2003**, *51*, 2496-2500, 10.1021/jf026247s.
10. Sylwia Mildner-Szkudlarz; Henryk H. Jeleń; The potential of different techniques for volatile compounds analysis coupled with PCA for the detection of the adulteration of olive oil with hazelnut oil. *Food Chemistry* **2008**, *110*, 751-761, 10.1016/j.foodchem.2008.02.053.
11. Tomas Cajka; Katerina Riddellova; Eva Klimankova; Monika Cerna; Frantisek Pudil; Jana Hajslova; Traceability of olive oil based on volatiles pattern and multivariate analysis. *Food Chemistry* **2010**, *121*, 282-289, 10.1016/j.foodchem.2009.12.011.
12. Diego L. García-González; Nalda Romero; Ramón Aparicio; Comparative Study of Virgin Olive Oil Quality from Single Varieties Cultivated in Chile and Spain. *Journal of Agricultural and Food Chemistry* **2010**, *58*, 12899-12905, 10.1021/jf1031313.
13. Benoit Berlioz; Christophe Cordella; Jean-François Cavalli; Louisette Lizzani-Cuvelier; André-Michel Loiseau; Xavier Fernandez; Comparison of the Amounts of Volatile Compounds in French Protected Designation of Origin Virgin Olive Oils. *Journal of Agricultural and Food Chemistry* **2006**, *54*, 10092-10101, 10.1021/jf061796+.
14. C. Pizarro; S. Rodríguez-Tecedor; N. Pérez-Del-Notario; José María González Sáiz; Recognition of volatile compounds as markers in geographical discrimination of Spanish extra virgin olive oils by chemometric analysis of non-specific chromatography volatile profiles. *Journal of Chromatography A* **2011**, *1218*, 518-523, 10.1016/j.chroma.2010.11.045.
15. Beatriz Quintanilla-Casas; Sofia Bertin; Kerstin Leik; Julen Bustamante; Francesc Guardiola; Enrico Valli; Alessandra Bendini; Tullia Gallina Toschi; Alba Tres; Stefania Vichi; et al. Profiling versus fingerprinting analysis of sesquiterpene hydrocarbons for the geographical authentication of extra virgin olive oils. *Food Chemistry* **2020**, *307*, 125556, 10.1016/j.foodchem.2019.125556.
16. Stefania Vichi; Lorena Pizzale; Lanfranco Conte; Susana Buxaderas; Elvira López-Tamames; Solid-Phase Microextraction in the Analysis of Virgin Olive Oil Volatile Fraction: Characterization of Virgin Olive Oils from Two

Distinct Geographical Areas of Northern Italy. *Journal of Agricultural and Food Chemistry* **2003**, *51*, 6572-6577, 10.1021/jf030269c.

17. Ouni Youssef; Flamini Guido; Issaoui Manel; Nabil Ben Youssef; Cioni Pier Luigi; Hammami Mohamed; Douja Daoud; Zarrouk Mokhtar; Volatile compounds and compositional quality of virgin olive oil from Oueslati variety: Influence of geographical origin. *Food Chemistry* **2011**, *124*, 1770-1776, 10.1016/j.foodchem.2010.08.023.
18. Arinç Kaftan; Yeşim Elmaci; Aroma Characterization of Virgin Olive Oil from Two Turkish Olive Varieties by SPME/GC/MS. *International Journal of Food Properties* **2011**, *14*, 1160-1169, 10.1080/10942910903453371.
19. Ioanna Kosma; Anastasia Badeka; Kornilia Vatavali; Stavros Kontakos; Michael Kontominas; Differentiation of Greek extra virgin olive oils according to cultivar based on volatile compound analysis and fatty acid composition. *European Journal of Lipid Science and Technology* **2015**, *118*, 849-861, 10.1002/ejlt.201500293.
20. Igor Lukić; Silvia Carlin; Ivana Horvat; Urska Vrhovsek; Combined targeted and untargeted profiling of volatile aroma compounds with comprehensive two-dimensional gas chromatography for differentiation of virgin olive oils according to variety and geographical origin. *Food Chemistry* **2019**, *270*, 403-414, 10.1016/j.foodchem.2018.07.133.
21. Nigri, S.; Oumeddour, R.; Fernandez, X.; Analysis of some Algerian virgin olive oil by headspace solid phase microextraction coupled to gas chromatography / mass spectrometry. *Riv. Ital. Delle Sostanze Grasse* **2012**, *84*, 54-61, .
22. Stefania Vichi; Josep M. Guadayol; Josep Caixach; Elvira López-Tamames; Susana Buxaderas; Monoterpene and sesquiterpene hydrocarbons of virgin olive oil by headspace solid-phase microextraction coupled to gas chromatography/mass spectrometry. *Journal of Chromatography A* **2006**, *1125*, 117-123, 10.1016/j.chroma.2006.05.029.
23. G. Luna; M.T. Morales; R. Aparicio; Characterisation of 39 varietal virgin olive oils by their volatile compositions. *Food Chemistry* **2006**, *98*, 243-252, 10.1016/j.foodchem.2005.05.069.
24. Francesca Blasi; Luna Pollini; Lina Cossignani; Varietal Authentication of Extra Virgin Olive Oils by Triacylglycerols and Volatiles Analysis. *Foods* **2019**, *8*, 58, 10.3390/foods8020058.
25. Eirini Pouliarekou; Anastasia Badeka; Maria Tasioula-Margari; Stavros Kontakos; Francesco Longobardi; Michael G. Kontominas; Characterization and classification of Western Greek olive oils according to cultivar and geographical origin based on volatile compounds. *Journal of Chromatography A* **2011**, *1218*, 7534-7542, 10.1016/j.chroma.2011.07.081.
26. Panagiota-Kyriaki Revelou; Charis Pappa; Eleni Kakouri; Charalabos D. Kanakis; George K. Papadopoulos; Christos S. Pappas; Petros A. Tarantilis; Discrimination of botanical origin of olive oil from selected Greek cultivars by SPME-GC - MS and ATR-FTIR spectroscopy combined with chemometrics. *Journal of the Science of Food and Agriculture* **2020**, *101*, 2994-3002, 10.1002/jsfa.10932.
27. F Angerosa; M Servili; R Selvaggini; A Taticchi; S Esposto; G Montedoro; Volatile compounds in virgin olive oil: occurrence and their relationship with the quality. *Journal of Chromatography A* **2004**, *1054*, 17-31, 10.1016/s0021-9673(04)01298-1.
28. Rocío Garrido-Delgado; María Del Mar Dobao-Prieto; Lourdes Arce; Miguel Valcárcel; Determination of volatile compounds by GC–IMS to assign the quality of virgin olive oil. *Food Chemistry* **2015**, *187*, 572-579, 10.1016/j.foodchem.2015.04.082.
29. Curtis M. Kalua; Danny R. Bedgood; Andrea G. Bishop; Paul D. Prenzler; Flavour quality critical production steps from fruit to extra-virgin olive oil at consumption. *Food Research International* **2013**, *54*, 2095-2103, 10.1016/j.foodres.2013.04.021.
30. Franca Angerosa; Influence of volatile compounds on virgin olive oil quality evaluated by analytical approaches and sensor panels. *European Journal of Lipid Science and Technology* **2002**, *104*, 639-660, 10.1002/1438-9312(200210)104:9/10<639::AID-EJLT639>3.0.CO;2-U.