Analysis of volatilome of olive oils and flavoured oils: quality grade evaluation and study of modification during storage

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The main aims of this research project are: i) setting up of chemometrics models based on volatilome analysis by HS-GC-IMS to predict olive oil commercial category, ii) evaluation of the qualitative and quantitative modification of volatile profiles of olive oils and flavoured oils during storage time. A total of 150 olive oil samples will be sensory evaluated and the volatile organic compounds of the same oils will be analyzed by HS-GC-IMS technique. The volatilome of a selected set of olive oils and flavoured oils will be studied by SPME-GC-MS during the storage time to register possible changes.

Analisi del volatiloma di oli di oliva e oli aromatizzati: valutazione della categoria merceologica e studio delle variazioni in conservazione

I principali obiettivi di questo progetto di ricerca sono: i) messa a punto di modelli chemiometrici per stimare la categoria merceologica dell'olio di oliva, ii) valutazione delle modifiche qualitative e quantitative dei profili in composti volatili di oli di oliva e di oli aromatizzati durante la conservazione. Un totale di 150 campioni saranno valutati sensorialmente ed i composti volatili degli stessi oli saranno analizzati mediante la tecnica HS-GC-IMS. Inoltre, il volatiloma di un set selezionato di oli di oliva e di oli aromatizzati sarà studiato attraverso SPME-GC-MS per registrare i possibili cambianti durante la conservazione.

# **1. State-of-the-Art**

In the Mediterranean area, especially in Spain, Italy and Greece, olive oil represents one of the main food products with a world production of 2,511,000 tonnes expected for the 2022/23 campaign (DG AGRI, 2023). In the European Union (EU), virgin olive oils (VOOs) can be classified into three commercial categories, based on both physicochemical and sensory parameters, such as: extra virgin (EV), virgin (V) and lampante (L) (Reg. EC n. 2022/2104). Despite several modifications that occurred over the years, the official method for sensory evaluation still shows some weaknesses as it is time-consuming and, in case of a non-correct training of assessors (Barbieri *et al*., 2020), can be affected by not satisfactory reproducibility of results. For this reason, the identification and quantification of volatile organic compounds (VOCs) in VOOs is of great importance for assessing their quality. In fact, targeted and untargeted instrumental methods, based on the analysis of such molecules, can be considered as an interesting tool useful to support the Panel test (Cavalli *et al*., 2004). Specifically, some VOCs have been proposed as markers to detect positive (e.g., fruity) and negative sensory attributes according to their concentrations (Valli *et al*., 2020). For this purpose, targeted methods based on headspace solid phase microextraction (SPME) with the use of flame ionization detector (FID) or mass spectrometry (MS) are being recently validated (Aparicio-Ruiz *et al*., 2023). Furthermore, rapid instrumental methods concerning gas-chromatographic techniques, such as Flash-GC and Ion Mobility Spectrometry (HS-GC-IMS) can be also useful for this aim permitting a fast pre-classification of samples and increasing the efficiency of quality control analyses (Valli *et al*., 2020). Finally, in the global economic scenario of olive oil, flavoured oils are becoming increasingly popular. Customers are attracted by their versatility of culinary use due to the possibility to convey a wide range of aromas to food preparations thanks to the use of different kind of flavouring matrices as herbs, spices, fruits, and vegetables (Baiano *et al*., 2016). The addition of specific flavouring agents to olive oils, depending on the applied technology to produce the flavoured oil (co-extraction, contact and essential oils inclusion), affects the incorporation in the oil matrix of specific bioactive compounds with antioxidant and/or healthy and/or sensory properties. Consequently, the analytical assessment of flavoured oils taking into consideration compositional and sensory characteristics is essential to check the product quality and to study the performance during storage (Lamas *et al*., 2022).

# **2. PhD Thesis Objectives and Milestones**

Within the overall objective mentioned above, this PhD thesis project can be subdivided into the following activities according to the Gantt diagram given in Table 1:

A1) **Sampling**: preparation, anonymization, and shipment of oils to the involved laboratories.

A2) **Definition of instrumental and sensory protocols:** setting up of analytical protocols for GC-IMS and sensory analysis to be applied in a shared mode by the five different laboratories.

A3) **Sensory and instrumental alignment tests**: verification of the degree of analytical alignment of five GC-IMS instruments using specific standards prepared ad hoc. The same analytical protocol has to be applied by the five laboratories participating in the trial (A3.1); check of sensory alignment among the five panels participating in the trial by application of a specific decision tree scheme (A3.2).

A4) **Creation of sensory and instrumental datasets:** the dataset will consist of at least 150 samples analysed by both sensory and instrumental analysis (GC-IMS).

A5) **Development of chemometric models:** estimation models (EV vs V) will be built using the dataset (A4).

A6) **Shelf-life study**: evaluation of the sensory characteristics and volatile profiles of selected samples (EV and V olive oils) monitored during the shelf-life (0, 6, 12 months) by SPME-GC-MS/FID and sensory descriptive analysis.

A7) **Volatile and sensory analysis of flavoured oils**: flavoured oils of particular interest for the company will be selected; volatilome and sensory characteristics as well as modification during storage will be monitored.

A8) **Writing and Editing** of the PhD thesis, scientific papers and oral and/or poster communications.

***Table 1***Gantt diagram for this PhD thesis project.

|  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Activity Months | | **1** | **2** | **3** | **4** | **5** | **6** | **7** | **8** | **9** | **10** | **11** | **12** | **13** | **14** | **15** | **16** | **17** | **18** | **19** | **20** | **21** | **22** | **23** | **24** |
| A1) | ***Sampling*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| A2) | ***Definition of instrumental and***  ***sensory protocols*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| A3) | ***Sensory and instrumental alignment tests*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 1) Instrumental alignment |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
|  | 2) Sensory alignment |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| A4) | ***Creation of sensory and***  ***instrumental datasets*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| A5) | ***Development of chemometric models*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| A6) | ***Shelf-life study*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| A7) | ***Volatile analysis of flavoured oils*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |
| A8) | ***Writing and Editing*** |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |  |

# **3. Selected References**

Agriculture and Rural Development (DG AGRI). DASHBOARD: OLIVE OIL, last update 17.03.2023: <https://agriculture.ec.europa.eu/system/files/2023-04/olive-oil-dashboard_en.pdf>

Aparicio-Ruiz R, Ortiz-Romero C, Casadei E, García-González DL, Servili M, Selvaggini R, Lacoste F, Escobessa J, Vichi S, Quintanilla-Casas B, Pierre-Alain G, Lucci P, Moret, E, Valli E, Bendini A, Gallina Toschi T (2022) Collaborative peer validation of a harmonized SPME-GC-MS method for analysis of selected volatile compounds in virgin olive oils. *Food Control* **135**: 108756.

Baiano A, Previtali M. A, Viggiani I, Varva G, Squeo G, Paradiso, V.M, Caponio F (2016) As oil blending affects physical, chemical, and sensory characteristics of flavoured olive oils. *Eur Food Res Technol*: **242**:1693-1708.

Barbieri S, Brkić Bubola K, Bendini A, Bučar-Miklavčič M, Lacoste F, Tibet U, Gallina Toschi T (2020) Alignment and proficiency of virgin olive oil sensory panels: The OLEUM approach. *Foods* **9:** 355.

Cavalli JF, Fernandez X, Lizzani-Cuvelier L, Loiseau AM (2004) Characterization of volatile compounds of French and Spanish virgin olive oils by HS-SPME: Identification of quality-freshness markers. *Food Chem* **88**: 151-157.

Commission Delegated Regulation (EU) 2022/2104 of 29 July 2022 supplementing Regulation (EU) No 1308/2013 of the European Parliament and of the Council as Regards Marketing Standards for Olive Oil, and Repealing Commission Regulation (EEC) No 2568/91 and Commission Implementing Regulation (EU) No 29/2012. OJ L **284**: 1–22.

Lamas S, Rodrigues N, Peres A.M, Pereira J.A (2022) Flavoured and fortified olive oils - Pros and cons. *Trends Food Sci* **124**:108-127.

Valli E, Panni F, Casadei E, Barbieri S, Cevoli C, Bendini A, García-González DL, Gallina Toschi T (2020) An HS-GC-IMS method for the quality classification of virgin olive oils as screening support for the panel test. *Foods* **9**: 657.