

PhD thesis and paper preparation and submission.

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PhD School of the University of Torino PhD Program in Agricultural, Forest and Food Sciences Largo Paolo Braccini 2 10095 Grugliasco (TO), Italy



Food lipids characterization: a tool for food quality assurance and authenticity

Nicolò Ivan Salgarella (<u>nicoloivan.salgarella@unito.it</u>) Department of Agricultural, Forest and Food Sciences, University of Turin, Turin, Italy Tutor: Prof. Vladimiro Cardenia, co-tutor: Dr. Manuela Giordano

The aim of the present PhD research project is to define a robust characterization and identification of the oils and fats ubiquitous used in food processing, either pure or in blends. Through the combination of different analytical techniques such as LC-HRMS, GC-MS and GC-FID, the fingerprint of the fatty substances, from raw materials to finished processed products, will be defined. The combination of these analytical techniques and chemometric data analysis, will be useful for identifying markers of technological processing (such as hydrogenation, interesterification and esterification) as related to commercial classification of the final products in term of quality and authenticity.

Caratterizzazione delle matrici grasse: uno strumento per il controllo della qualità e l'autenticità

Il campo applicativo del presente progetto di dottorato è quello delle sostanze grasse ampiamente impiegate nell'industria alimentare, in particolare nell'industria dolciaria. Lo scopo di questo progetto è quello di definire una corretta caratterizzazione ed identificazione delle componenti delle sostanze grasse per delinearne la qualità e autenticità. Mediante la combinazione di tecniche analitiche quali LC-HRMS, GC-MS e GC-FID verrà condotta una caratterizzazione puntuale delle sostanze grasse di interesse (dalle materie prime fino al loro impiego nei prodotti finiti) al fine di ottenere un quadro completo della composizione (*fingerprint*). Mediante la combinazione di tecniche analitiche performanti e dell'analisi chemiometrica dei dati si identificheranno i possibili *markers* di processo (ad es. idrogenazione, inter-esterificazione ed esterificazione) utili alla definizione di qualità e autenticità delle materie grasse di interesse industriale.

STATE OF ART

The guarantee of authenticity and quality along the food chain is demanding great attention from the food industry and consumer. In the recent years, the food manipulation is undergoing radical changes that requires a deeper characterization of the food chain, starting from raw materials up to the final products.

The characterization of fats in processed products such as spreads, bakery products and other confectionery products, is important for consumer point of view and to contrast the spread of incorrect or fraudulent food handling techniques that are often still not regulated by law (Ben-Ayed et al., 2013).

Oils and fats are the main components of food and processed food products. The fats in foods play a key role as related to their diffusion properties, hardness and palatability. Crystallization and melting behavior of fats are important quality factors in various products, such as in confectionery and pastry (Santoro et al., 2018). Again, plastic and structural properties of fats influenced some crucial sensory parameters like texture, spreadability and mouthfeel linked to networks of fat crystals (Marangoni, et al., 2020). Dietary fats and oils are complex matrices containing a large and different variety of compounds such as triacylglycerols (TAG), diacylglycerols (DAG), monoacylglycerols (MAG), free fatty acids (FFA), phospholipids and other minor components such as sterols (Behrman & Gopalan, 2005). Major and minor components in fat matters are crucial for their characterization; the presence or absence of individual components is useful for establishing the oil purity and for detecting the application of certain processing conditions. The study of positional triglycerides (Santoro et al., 2018), *trans* fatty acids profile, sterol composition, small molecules as dialkyl ketones (Santoro et al., 2018) and oxidized sterols profile in vegetable and in dairy fat fractions (Risso et al., 2021) can set reference points in the evaluation of technological processes (lawful or unlawful) adopted in the supply chain. Information contained is often too much and important predictor signals are overlooked by the common analyzes. Chemometrics will be a potential tool for the evaluation of multivariate chemical data and useful for identifying processing markers (van Ruth et al., 2010). In addition, the multivariate data analysis methods will be used to build a statistic model ables to accurately predict the mentioned characteristics that are not easy to identify in order to rebuild the background (Kamal & Karoui, 2015).



- A. Development of analytical methods to study the saponifiable and unsaponifiable fractions of fats. Gas chromatographic analysis (GC-FID) will be applied for defining the fatty acids, triacylglycerols, mono and diacylglycerols composition of different complex fats and oils. Interlaboratory tests will be set up to evaluate robustness, sensitivity and reproducibility of developed methods. In order to reach a better knowledge on fat composition, especially for minor components, both the gas chromatography and liquid chromatography (HPLC) coupled to low and high-resolution (HRMS) will be tested. In particular, by mean of the use of HRMS techniques, the untargeted compounds will be defined and high specificity on the recognition of targeted compounds will be reached.
- B. Collection data. Raw materials such as palm oil, palm kernel oil, cocoa butter oil, rice oil, sal oil, shea oil, coconut and other mainly used tropical oils as well as their commercial blends will be analyzed. Raw and processed samples already extracted by common extraction and refining techniques / modification techniques will be analyzed.
- C. Development of chemometrics methods database analysis. Principal Components Analysis (PCA), Discriminant Analysis (DA) and Partial Least Squares Discriminant Analysis (PLS–DA) models are most promising techniques.
- D. Study of the confectionery industry product. Fat matter of commercial samples from the confectionery industry will be extracted and investigated. Database and chemometrics tool will be used for careful product recognition.
- 3[#] Year 2[#] Year 3 5 9 11 13 15 1 2 4 6 7 8 10 12 14 16 17 18 19 20 21 22 23 24 Development of analytical methods to study the saponifiable and Α unsaponifiable fractions. В Collection data. **Development of chemometrics** С methods – database analysis. Study of the confectionery industry D product. PhD thesis and paper preparation Ε and submission. -

References



Ben-Ayed, R., Kamoun-Grati, N. and Rebai, A. (2013). An overview of the authentication of olive tree and oil. *Comprehensive Reviews in Food Science and Food Safety*, *12*(2), 218–227. Kamal, M. and Karoui, R. (2015). Analytical methods coupled with chemometric tools for determining the authenticity and detecting the adulteration of dairy products: A review. *Trends in Food Science and Technology*, *46*(1), 27–48. Lee, D. S., Noh, B. S., Bae, S. Y. and Kim, K. (1998). Characterization of fatty acids composition in vegetable oils by gas chromatography and chemometrics. *Analytica Chimica Acta*, *358*(2), 163–175. Marangoni, A. G., Van Duynhoven, J. P. M., Acevedo, N. C., Nicholson, R. A. and Patel, A. R. (2020). Advances in our understanding of the structure and functionality of edible fats and fat mimetics. *Soft Matter*, *16*(2), 289–306. Risso, D., Leoni, V., Fania, C., Arveda, M., Falchero, L., Barattero, M. Barattero, A. Civra, D. Lembo, G. Poli, G. and Menta, R. (2021). Effect of industrial processing and storage procedures on oxysterols in milk and milk products. *Food and Function*, *12*(2), 771–780. Santoro, V., Baiocchi, C., Dal Bello, F., Gastaldi, D., Aigotti, R., Zorzi, M., and Medana, C. (2018). Formation of by-products during chemical interesterification of lipids. Detection and characterization of dialkyl ketones by non-aqueous reversed-phase liquid chromatography-high resolution mass spectrometry and gas chromatography-mass spectrometry. *Journal of Chromatography A*, *1581–1582*, 63–70. Santoro, V., Dal Bello, F., Aigotti, R., Gastaldi, D., Romaniello, F., Forte, E. and Medana, C. (2018). Characterization of the identity of fats and oils by their fatty acid, triacylglycerol and volatile compositions using PLS-DA. *Food Chemistry*, *118*(4), 948–955.