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Review

# Chemometric applications to assess quality and critical parameters of virgin and extra-virgin olive oil. A review

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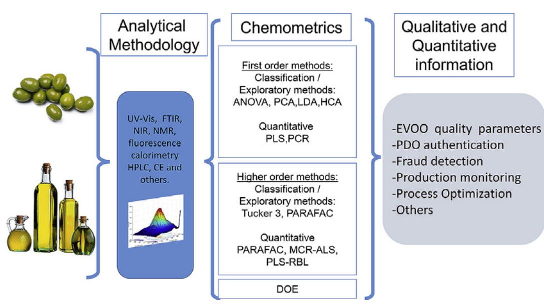
## HIGHLIGHTS

- First work review addressing chemometrics coupled to almost all analytical methods used for EVOO/VOO.
- Discussion on some practical aspects about implementing chemometrics on EVOO/VOO.
- Screening a wide range of techniques used for EVOO/VOO analysis.
- Perspective of the application of chemometrics in EVOO/VOO analysis in the coming years.

## GRAPHICAL ABSTRACT

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## ABSTRACT

Today virgin and extra-virgin olive oil (VOO and EVOO) are food with a large number of analytical tests planned to ensure its quality and genuineness. Almost all official methods demand high use of reagents and manpower. Because of that, analytical development in this area is continuously evolving. Therefore, this review focuses on analytical methods for EVOO/VOO which use fast and smart approaches based on chemometric techniques in order to reduce time of analysis, reagent consumption, high cost equipment and manpower.

Experimental approaches of chemometrics coupled with fast analytical techniques such as UV–Vis spectroscopy, fluorescence, vibrational spectroscopies (NIR, MIR and Raman fluorescence), NMR spectroscopy, and other more complex techniques like chromatography, calorimetry and electrochemical techniques applied to EVOO/VOO production and analysis have been discussed throughout this work. The advantages and drawbacks of this association have also been highlighted.

Chemometrics has been evidenced as a powerful tool for the oil industry. In fact, it has been shown how chemometrics can be implemented all along the different steps of EVOO/VOO production: raw material input control, monitoring during process and quality control of final product.

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**Contents**



## 1. Introduction

Among vegetable oils, virgin olive oil (VOO) and extra-virgin olive oil (EVOO) have nutritional and sensory characteristics that make them unique and basic components of the Mediterranean diet. Olive oil has been used over the centuries for its preventive and therapeutic properties, as well as a precious and valuable dietary lipid ingredient [1]. In fact, compared to other vegetable oils normally subjected to a refining process, EVOO is well-known for having higher quality in terms of health and sensory aspects, and because of its characteristic oxidative stability. These properties are related not only to the fatty acid composition of its lipid matrix, but especially to the presence of several minor compounds. Among these minor compounds there are:

- volatile compounds, mainly related to typical odours;
- phenolic compounds, responsible for its oxidative stability, taste and healthy properties;
- tocopherols, that contribute to oxidative stability and to healthy proprieties;
- squalene, related to their healthy proprieties

In the last three decades, many efforts have been made in order to correlate the chemical structures of these naturally occurring molecules in EVOO/VOO with its quality and genuineness, health benefits and sensory characteristics [2]. Other important aspect is the fluctuation of EVOO/VOO components (macro and micro) due to factors of different origins [3]. Some of these factors are listed below:

- agronomic variables (environment conditions, agricultural and harvesting methods, olive cultivar, olive ripening stage during the transformation phase)
- technological variables (milling, malaxation and separation of phases)
- storage/distribution parameters (time, temperature, light, packaging)

Due to its quality and genuineness, it is important to underline that EVOO is the most expensive edible oil. For economic reasons, it may be adulterated by the addition of cheaper oils such as refined olive oil, residue oil, synthetic olive oil-glycerol products, seed oils and nut oils. To avoid this kind of practice, a rapid method is important for quality control and labeling purposes [3].

The study of the different molecules present in EVOO/VOO has required the use of different sample preparation methodologies and analytical techniques. Among these techniques can be listed spectrometric techniques and separation techniques coupled to different detection systems, as diode array detection (DAD), mass spectrometry (MS) and nuclear magnetic resonance (NMR). In recent years, also chemometrics has been coupled to these analytical techniques as can be seen in Fig. 1.

The analyses performed by the different analytical tools provide a huge amount of data which most of the times, is difficult to process or poorly exploited. Usually, researchers applied univariate analysis to their data as analysis of variance (ANOVA), t-tests for normally distributed data, etc. However, developments in computer science have allowed the extensive use of multivariate procedures. This important step has made possible to cope with the vast amount of data that new and sophisticated analytical instruments provide. The application of mathematical algorithms has allowed to reach conclusions that some years ago were unthinkable [4].

Some reviews works have collected applications of some analytical techniques combined with multivariate analysis to authenticate, detect adulteration and determine intrinsic quality parameters in edible fats and oils. However, they are not at all doomed to the EVOO/VOO analysis or showed only partial point of view of EVOO/VOO analysis (only one or two analytical techniques coupled to chemometrics were revised) [5–9].

Nowadays, chemometrics is increasingly being applied in food analysis. In the VOO and EVOO field specifically, chemometrics is helping to find cultivar markers, to differ samples by cultivar/origin, to guarantee the authenticity of olive oil and/or detect adulteration with other oils.

Despite all described above, chemometrics has not already been considered by EU Regulation (EC No 2568/1991 Regulation and amendments), International Olive Council (IOC) trade standard (Trade standard applying to olive oils and olive-pomace oils, COI/T.15/NC No 3/Rev. 10 November 2015) nor USA Regulation (United States standards for grades of olive oil and olive-pomace oil) as a reliable tool for EVOO/VOO data treatment and quality assessment. Therefore, the potential of chemometrics should be highlighted and, thus, the aim of this review is to summarize the main applications of chemometrics coupled to the most important analytical techniques for the analysis of VOO and EVOO in the last 20 years.

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