# Accelerated shelf life studies of extra virgin olive oils using the Oxitest method

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- The VELP Scientifica Oxitest is an analytical instrument that can be used to examine fat oxidation in whole samples.
- The Oxitest method was used to analyze the oxidative stability of extra virgin olive oils coming from two regions of Italy.
- There was a strong correlation between the total quantity of polyphenols and oxidation stability, notwithstanding differences in cultivation and geographic origin.

In the food industry, it is very important to determine the shelf life of raw materials and processed foods as quickly as possible. The Oxitest method is a rapid analytical technique used to examine fat oxidation. The method's key advantage is that results are obtained by analyzing whole samples, whereas other techniques require the fat content to be extracted before oxidation tests are performed. The Oxitest method has been recognized as an AOCS International Standard Procedure (Cd 12c-16) thanks to VELP Scientifica's efforts to develop the instrument and method in collaboration with universities and research centers. The new method is in the *Official Methods and Recommended Practices of the AOCS*, 7th Edition, and can be preordered at https://www.aocs.org/store/shop-aocs/shop-aocs?productId=72560532.

Lipid oxidation is influenced by a variety of factors, including light, heat, fatty acid composition and degree of saturation, the form and concentration of oxygen, and the presence of minor compounds such as antioxidants, metals, and pigments. Systematic studies on these factors have been conducted to improve the oxidative stability of foods (Comandini *et al.* 2009). Lipid oxidation is a slow process that could last days or months, depending on the sample. The Oxitest reactor subjects samples to a high-oxidative-stress environment, so that their resistance to oxidation can be evaluated in a short period of time (Comandini *et al.* 2009).

# LIPID OXIDATION



The instrument features two thermostated and hermetically sealed titanium chambers in which oxygen is purged until the pressure within both chambers is between 0–8 bar (Fig. 1). The temperature is then set to the desired level (room temp. to 110 °C). The Oxitest measures the absolute pressure change inside the two chambers monitoring the oxygen uptake of the active components of the samples, and automatically generates a value expressed in time, called the Induction Period (IP). The longer the IP, the more resistant a sample is to oxidation over the life of the sample.

During the past several years, shelf life studies of vegetable oils have raised considerable attention, and several scientists have started investigating how vegetable oils respond to oxidation tests. Extra virgin olive oil has become an increasingly popular subject of such studies, as this particular oil is an important component of the Mediterranean diet and is broadly recognized for its positive health benefits.

Lipid oxidation is the main degradation process affecting the shelf life and compromising the organoleptic and nutritional characteristics of oils, fats, and foods that contain them. (Tura *et al.* 2007). Olive oil has more oxidative stability than other vegetable oils due to its low unsaturated fat content (Martinez *et al.*, 2014). Extra virgin olive oils are particularly resistant to oxidation due to the high quality of raw material and softer method of extraction. In fact, extra virgin olive oils have a higher percentage of natural antioxidants, particularly of polyphenols and  $\alpha$ -tocopherol (Favati *et al.* 2013).



FIG. 1. Sample loading in the Oxitest chambers

# **Further reading**

Comandini, P., V. Verardo, P. Maiocchi, and M.F. Caboni, Accelerated oxidation: Comparative study of a new reactor with oxidation stability instrument, *Eur. J. Lipid Sci. Technol. 111*: 933–940, 2009

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Scientists from the University of Basilicata (Potenza, Italy), the University of Verona, and VELP Scientifica (Usmate, Italy) have conducted stability tests on extra virgin olive oil as a part of the "Eufolia Mediterranea" project, aiming to assess the behavior under oxidative stress of extra virgin oils from conventional and experimental olive cultivation. Three samples from the cultivar *Ogliarola del Bradano* were obtained from the same field in the Basilicata region in southern Italy. All three samples were extracted in the same facility under the



### A) Basilicata Oils

same operative conditions. The oxidation stability tests were performed at a constant temperature of 90°C and an oxygen pressure of 600 kPa, with three repetitions for each sample.

The three samples differed with respect to the year and the method in which the olives were cultivated. The first sample (PFI 2013 C) was obtained in 2013, from a conventional cultivation. The second (PFI 2014 C) was also obtained from a conventional cultivation, but in the following year (2014). The third sample (PFI 2014 E), also obtained in 2014, was obtained from an experimental cultivation in which low environmental impact and high-efficiency technologies were used.

When the IP values of the three investigated oils were compared, the oil from conventional cultivation in 2013 had a higher oxidative stability than the oil that was conventionally cultivated in 2014, which was likely due to climatic parameters. However, the sample obtained from the experimental cultivation in 2014 was considerably more stable, with an IP value 10 h longer than the sample obtained from conventional cultivation during the same year, indicating that the experimental cultivation techniques had a positive effect on the oxidative stability of extra virgin olive oil.

The oxidative stability of the three oils from the Eufolia Mediterranea project was compared with the oxidative stability of other oils from the Basilicata region, including two oils from the same variety (*Ogliarola del Bradano*), one oil from the variety *Leccino*, and another one from the *Coratina* variety. Among the samples tested, those from the *Leccino* variety had the lowest IP value (19.5 h), and those from *Coratina* the highest (62 h).

Moreover, the oxidative stability of five PDO (Protected Designation of Origin) extra virgin olive oils from the Garda lake area in northern Italy, was analyzed. These oils were characterized by IP values ranging from 38 to 78 hours

#### B) Garda DOP Oils



FIG. 2. Correlation between IP values and total polyphenol content for Basilicata(a) and Garda(b) extra virgin olive oils

The IP assessed values for all the investigated oils span over a wide range, namely from 20 to 78 hours, with an observed overlapping of the specific ranges of the Basilicata and the Garda oils, showing anyhow the latter the highest IP values. However, the available analytical data did not allow to highlight a possible direct correlation between the geographical origin and IPs. In the study the extra virgin oils were also characterized taking into account other parameters: acidity, peroxides, K232, K270, p-Anisidine, chlorophyll, carotenoids, tocopherols, and total polyphenols. The results showed that when considering oils having the same geographical origin it was possible to highlight a significant positive correlation between the total polyphenols content and the assessed IPs, with R<sup>2</sup>> 0.993 and R<sup>2</sup>> 0.984 for Basilicata and Garda oils, respectively (Fig. 2). Conversely, such a strong correlation could not be observed for the other investigated parameters.

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