Olive Oil Analysis Explained – Part I: Basic Assays

International trade standards specify that virgin olive oil is extracted exclusively from olives, solely by mechanical means, without excessive heating or any chemical processing. Virgin olive oil is a natural product that does not need much to be preserved. It contains antioxidants, Nature's preservatives that extend the oil's shelf life if stored in a sealed bottle, away from the light and in cool temperature.

Compared to wine making, olive oil production is a simpler and faster process. But unlike wine, olive oil does not age gracefully; it is at its best when fresh, and will unavoidably deteriorate over time. Yet the volatile components that give olive oil its desirable "green notes", the flavor and aroma that consumer and tasters desire, will gradually disappear with storage.

Virgin olive oil is a natural product with anti-oxidants, Nature's preservatives. Yet, desirable flavor, aroma and 'green notes' will gradually disappear.

Three basic laboratory assays are required to determine the oil's grade. They measure olive oil's attributes related to quality of the olive fruit and its processing, and assess the oil's present state. Basic assays are essential for producers to assess the quality, freshness and potential shelf life of their oil, and are also of interest to buyers. Buyers will also be concerned with detecting possible oil adulteration, the topic for subsequent article.

Here, focusing on issues that concern olive oil producers, we will describe the natural processes that affect olive oil quality and the meaning of the basic analytical results.

From olive fruit to bottled oil

Superior olive oil of extra virgin grade is obtained only when harvest, milling and processing of olives is properly managed. Along those steps, there are two naturally occurring and inter-related processes that must be understood as they impact the olive oil's quality: *lipolysis* and *oxidation*.

Lipolysis *(*a form of *hydrolysis)* begins on the olive fruit as it ripens, and is caused by *enzymes* present in the fruit, which are later on removed with the *vegetation water*. *Lipolytic* enzymes break down major olive oil components and generate *free fatty acids,* precursors to olive oil deterioration.

Oxidation (more specifically, *auto-oxidation)* is triggered when the oil enters in contact with oxygen in the air, first during the milling process, and later on during storage in tanks and bottles. Oxidation produces *peroxides* from some fatty acids. Peroxides are unstable compounds which are further oxidized to yield volatile and non-volatile components that give rise to off-flavors and undesirable aromas in the oil *(secondary oxidation)*. The other form of oxidation, *photo oxidation*, is usually minor and negligible if the oil is stored in the dark.

In brief: lipolysis generates free fatty acids and oxidation causes the formation of peroxides from these fatty acids (*primary oxidation*), leading to rancidity and degradation of the oil over time (*secondary oxidation*).

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Attaining olive oil of superior quality requires minimizing the generation of free fatty acids by hydrolysis and delaying the onset of oxidation.

Superior quality olive oil is attained by minimizing free fatty acids due to hydrolysis and delaying oxidation.

The oxidation process in oil
Oxidation process in oil takes place in three stages: *initiation, propagation* and *termination*.

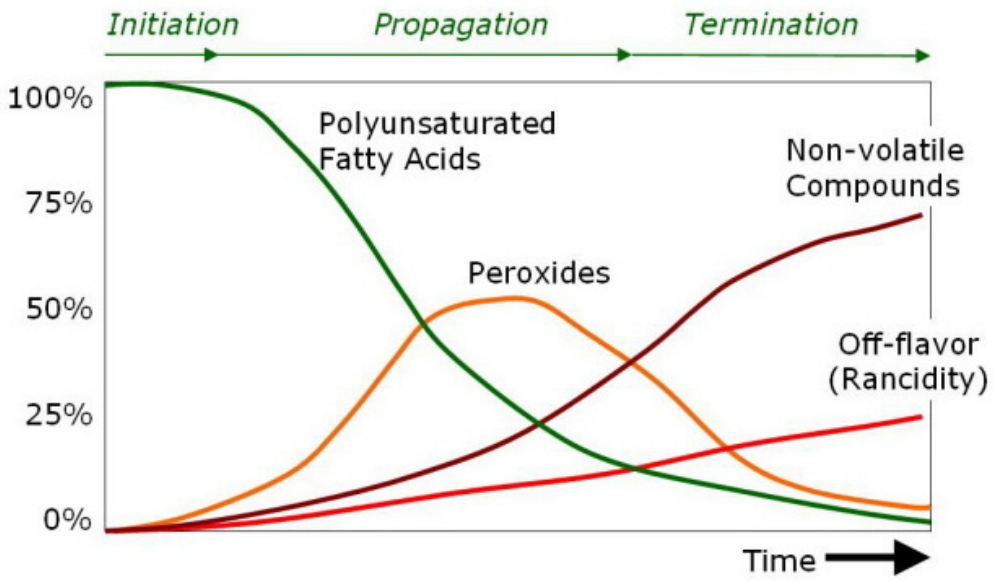

Figure 1: Olive Oil oxidation[1]

Figure 1 shows how the oxidation of fatty acids in olive oil changes with time. Once the oil has been decanted or centrifuged, the free fatty acid level (or *free acidity*) will gradually decline while the oil remains in storage. [*Declining free acidity is represented in the graph by the down-sloping line from upper-left to lower-right.*]

During the *initiation* stage, free fatty acids present in the oil become slowly oxidized, giving rise to small amounts of peroxides. Since peroxides, in turn, decompose into other substances, the overall peroxide level does not increase rapidly.

However, when certain levels of peroxides are reached in the oil, a *propagation* stage ensues during which peroxide levels will rapidly increase, as peroxides beget other peroxides. [*Increasing accumulation of peroxides is represented in the graph by the rising left-side of the curve.*]

During the *termination* stage, peroxides further decompose giving off numerous off-flavor compounds that humans can detect, even if present at extremely low concentrations—below parts per million. Therefore, even when just a tiny fraction of peroxides degrade into undesirable compounds, the oil will taste rancid. Even oils in late *initiation* stage of oxidation may already show signs of rancidity.

[1] Adapted from "Olive Oil Oxidation" by Maria Teresa Morales and Roman Przybylski. In "Handbook of Olive Oil, Analysis and Properties", John Hardwood and Ramon Aparicio, Editors (First edition, 1999)

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[*Rancidity is represented by the lower line rising from the bottom-left to the right of the graph.*].

If the quality of the olive fruit was very good and free acidity levels were low when processing the olives, there may be a one or two-year lag time before peroxide levels in the oil become problematic (that is, the initiation period is long). But if the olive fruit was spoiled, the temperature during processing high or the *malaxation* time too long, there is an increased risk of reaching higher peroxide levels much sooner, thereby shortening the oil's shelf life. [*Peroxides' evolution is represented by the bell-shaped curve in the center of the graph.*]

Olive Oil Quality Attributes

The basic olive oil parameters measured analytically are: Free Fatty Acids (FFA), Peroxide Value (PV) and UV *absorbance*. In combination, they give hints of proper or inadequate handling of the olive fruit prior to milling and assess the oil's present state of oxidation.

Free Fatty Acids (FFA) or Free Acidity
As mentioned earlier, free fatty acid formation precedes most, if not all oil deterioration. Free acidity levels increase by hydrolysis of the major oil molecules in the fruit (*triglycerides)* early in the production process, from harvest through milling, while water and plant enzymes are still in contact with the oil. For this reason, milling the olives soon after harvest, and promptly separating oil from vegetation water are critical to maintaining a low initial free acidity level, essential for higher-quality oil.

Free acidity values provide an indication of how the fruit was handled prior to processing and the length of time from harvest to milling. Free acidity is also an early indicator of the potential longevity of the oil. Higher quality oils recently produced, will exhibit very low acidity—it may be no higher than 0.35% in the best extra virgin olive oils (EVOO).

Peroxide Value (PV)
Peroxides, which are flavorless, are generated from the oxidation of polyunsaturated fatty acids in the oil (*linoleic* and *linolenic* acids). Peroxides are unstable, usually building up slowly and leading to oil rancidity. The likelihood of peroxides becoming rampant increases during storage. The onset of this decay process may be delayed for one or two years in good olive oil, rich in radical-scavenging antioxidants. But once peroxides are present, rancidity is unavoidable.

Peroxide Value identifies the early stages of oxidation. Subsequent oxidation is detected through UV absorbance and by the oil's sensory properties, such as rancidity, which result from the peroxides' breakdown.

To reduce peroxide levels and therefore delay oxidation of high-peroxide oils, blending may be a viable recourse. Otherwise, refining is the only option to eliminate peroxides and remove off-flavor substances. Obviously, the resulting product will not be virgin oil.

For premium quality, low initial Free Acidity is essential, while Peroxide Value identifies early stages of oxidation.

Ultraviolet (UV) Absorbance
UV tests determine the ultraviolet light *absorbance* measured by shining UV light through the oil at several specific wavelengths. Absorbance at K232 nm (*nanometers*), K270 nm and *Delta (Δ)*

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K [2]correlate with the state of oxidation by detecting specific oxidized compounds, some generated from secondary oxidation, and also detect possible adulteration with refined oils.

Phenols and antioxidants
The potential for olive oil to naturally deteriorate and become rancid is inherent in its chemical nature, since the unsaturated fatty acids in the oil are susceptible to breakdown and oxidation. But olive oil also has its own natural preservatives, antioxidants, which dampen the auto-generation of peroxides and increase the oil's shelf-life. We will cover this topic in part II.

Interpreting Analysis Results

The standard set of quality tests for grading olive oil includes *organoleptic* (sensory) tests and the three basic analyses: FFA, PV and UV. Though not included among them, the Total Phenol value is also regarded as a valuable quality measurement of the oil's potential stability, in addition to its health benefits.

The International Olive Committee's (IOC) olive oil standards specify the precise measurement thresholds to be met for oils to be graded as extra virgin, virgin, and so on. Whenever analytical parameters (FFA, PV, and UV) exceed a given threshold, the oil is classified as the next lower quality grade. But experts agree that a quality EVOO can meet more stringent thresholds than the standard, reflecting the freshness and quality of truly superior oil.

*Premium quality olive oil will show **Free Fatty Acids** lower than 0.3%, **Peroxide Value** less than 7 meq/kg, **K232** below 1.85, and no-sensory defects.*

The standard FFA threshold is 0.8% acidity, but fresh, high-quality EVOO can certainly achieve FFA values of 0.3 % or lower.

Basic assays assess the quality, freshness and potential shelf-life of olive oil, and are of equal interest to producers and buyers.

Peroxide Values vary widely over time. Some oils may meet the EVOO's 20 meq/kg threshold while already showing early signs of rancidity.

Experience indicates that high-quality, recently milled oils exhibit peroxide values below 12 meq/kg. Truly excellent oils may have PV as low as 7 meq/kg (9 meq/kg for organic oil). In the PV, the more likely the oil's shelf life will be extended.

Regarding UV absorbance, though the standards call for K232 below 2.5 and K270 below 0.22, superior olive oil will exhibit K232 values below 1.85 (2 for organic oil) and K270 below 0.17. As mentioned before, low values correlate with high-quality oil, as UV absorbance detects early and later states of oxidation.

Conclusion
A basic set of analysis provides producers and buyers with valuable information about the quality of fruit and processing that went into the oil and the state of subsequent natural deterioration (oxidation) that the oil has undergone.

Parts II and III of these papers will respectively describe polyphenols and organoleptic properties of oil, and olive oil purity and variety identification.

[2] The absorbance at a specific wavelength is conventionally referred to by K.

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