

# Evaluation of olive oil quality grade using a portable battery operated sensor system

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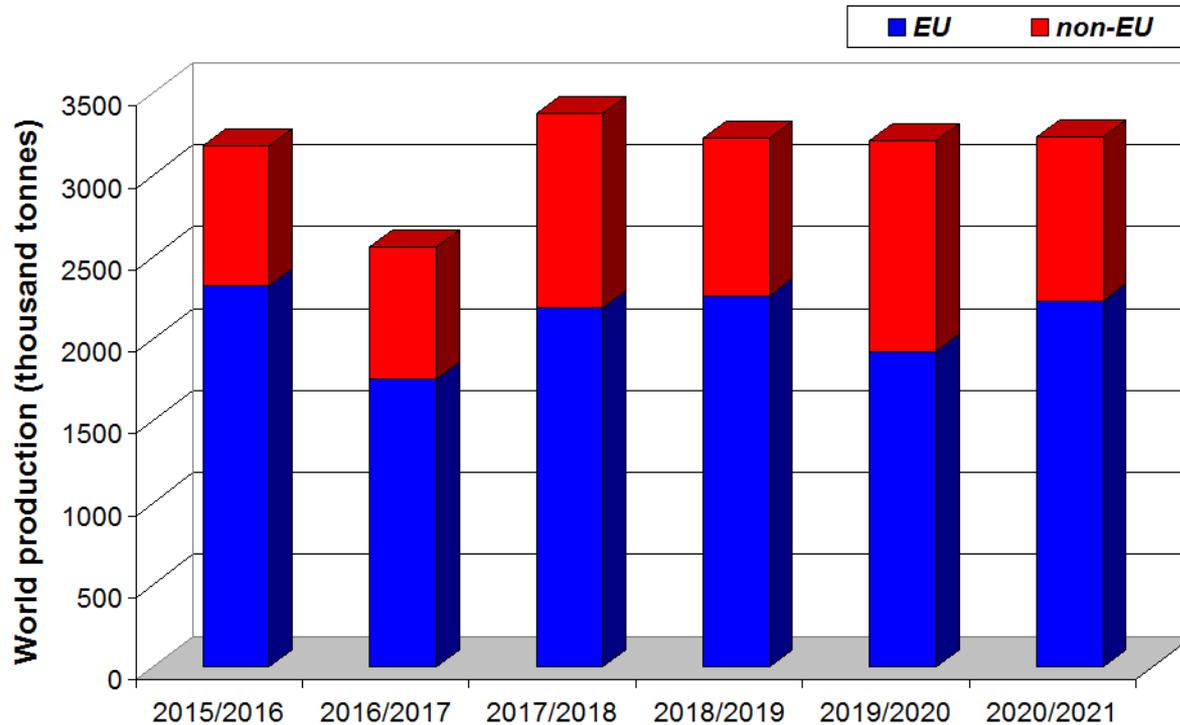
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# Olive oil health benefits

EVOO is responsible for many health benefits associated to Mediterranean diet as it is a fundamental ingredient of this diet.

- Reduction of microbial activity
- Anti-tumor properties
- Benefits for blood pressure
- Prevent diabetes
- Prevent osteoporosis
- Prevent neurodegenerative diseases
- Anti-inflammatory properties
- Benefits for the digestive system
- Prevent oxidative stress

# Olive oil market in the world

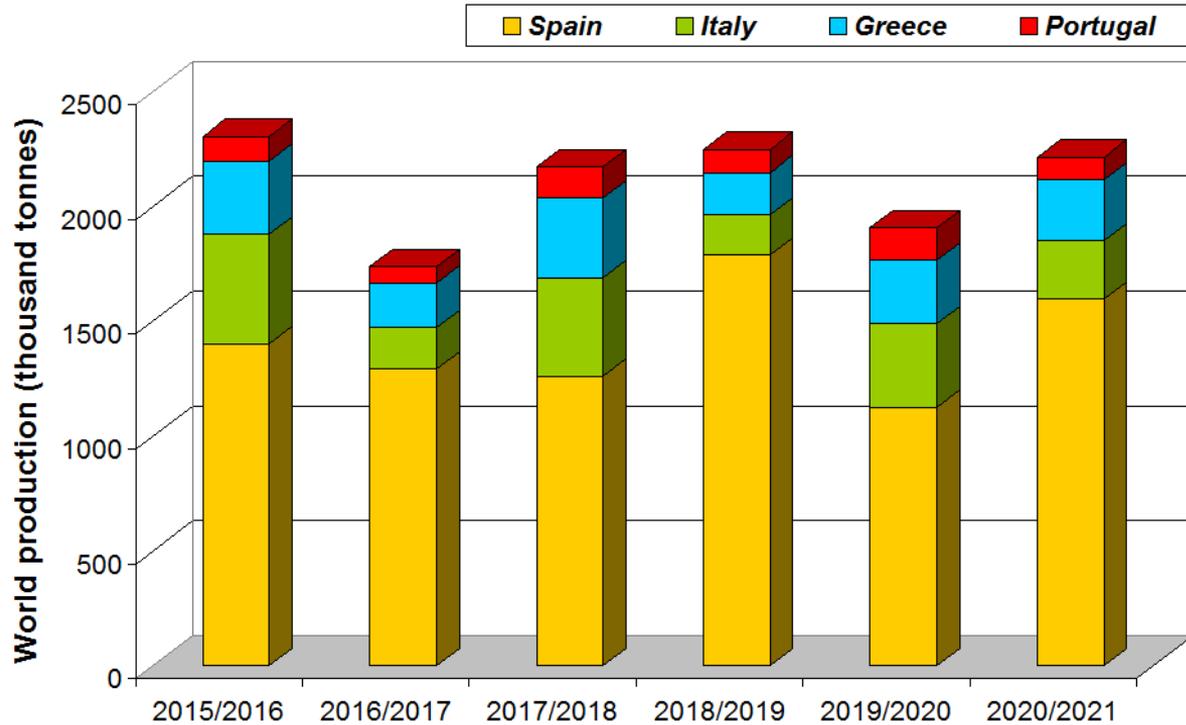


European Union (EU) is the largest olive oil producer in the world (about 66% of the world production).

EU is also the biggest olive oil consumer (1.5 million tonnes) and exporter (570000 tonnes).

Source: *International Olive Council*

# Olive oil market in the European Union



Spain is the biggest olive oil producer in EU and Italy is the second biggest producer.

Spain: 66%  
Italy: 15%  
Greece: 13%  
Portugal: 5%

Source: *International Olive Council*

# Olive oil quality

Olive oil is graded to different categories according to quality parameters:

## 1) Physico-chemical characteristics

- **Free acidity (FA):** amount of fatty acids no longer linked to their parent triglyceride molecules. It is affected by the olive quality and production process. It does not change significantly during storage.
- **Peroxide index (PI):** indicator of the oil primary oxidation. If storage conditions are not adequate, oil oxidation takes place and degrades the product quality.

## 2) Organoleptic characteristics

- **Sensory analysis** to check the absence of organoleptic defects.

# Olive oil quality

Based on the values of the different quality parameters, virgin olive oil can be classified into the following categories:

- **Extra Virgin Olive Oil (EVOO):** FA < 0.8%, PI < 20 meq O<sub>2</sub> / kg oil. From an organoleptic point of view, it has no defects and is fruity.
- **Virgin Olive Oil (VOO):** FA < 2%, PI < 20 meq O<sub>2</sub> / kg oil. It may have organoleptic defects at very low level.
- **Lampante Olive Oil (LOO):** FA > 2% and/or PI > 20 meq O<sub>2</sub> / kg oil. It has no fruity characteristics and substantial organoleptic defects. Lampante olive oil is not intended to be marketed at retail stage.

# Measurement of free acidity and peroxide index

The **reference techniques** to measure olive oil free acidity and peroxide index are **manual titration procedures**.

- The analysis must be carried out by trained personnel in a laboratory.
- Chemicals used in the titration must be properly disposed according to regulations.
- Small production centers, that can not afford an internal laboratory for quality analysis, must ship the samples to an external laboratory with high costs and long response time.

# Objective of the research project

The objective of the research project is the development of an **electronic instrument for the evaluation of quality grade of olive oil**. The instrument must have the following characteristics.

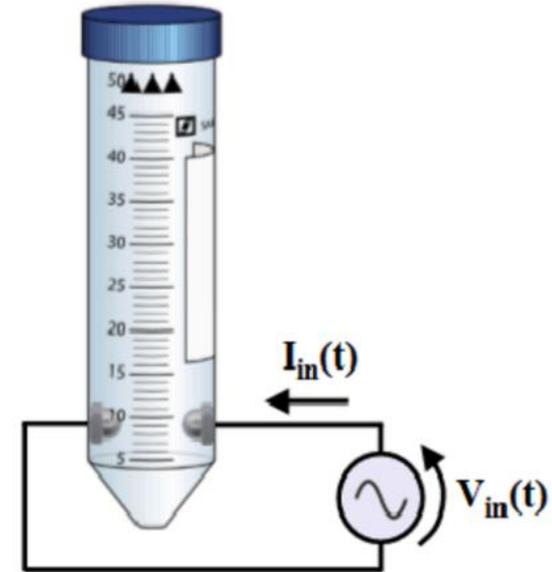
- It must be **portable** and **powered by batteries** to allow **in-situ measurements** in the production centers (oil mills and packaging centers).
- It must be **simple to use**. No particular skills for the operators.
- The measurement must be **quick**.
- **No toxic compounds** must be used.

# Working principle of the proposed technique

The working principle of the proposed technique is based on the **measurement of the electrical characteristics** of an emulsion between an **hydro-alcoholic solution** (60% ethanol, 40% distilled water) and the **olive oil under test**.

The emulsion is prepared by mixing **15 mL of hydro-alcoholic solution** and **1 mL of the olive oil** under test.

The emulsion is stored in a 50 mL Falcon vial modified to feature **a couple of stainless steel electrodes** (6 mm diameter, spaced by 12 mm).



# Working principle of the proposed technique

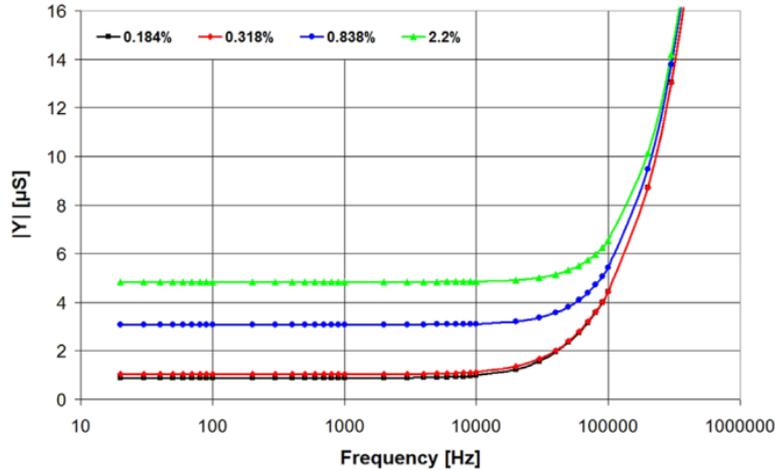
- Preliminary measurements have been carried out in a controlled laboratory environment on four olive oil samples with different levels of free acidity.
- The emulsion is stored in a thermal chamber with operative temperature 20 °C.
- The emulsion electrical characteristics have been analyzed by Electrical Impedance Spectroscopy (EIS) in the frequency range 20 Hz – 2 MHz using a commercial impedance analyzer.

# Working principle of the proposed technique

- From an electrical point of view, the emulsion can be modeled as the parallel of an electrical conductance  $G_m$  and an electrical capacitance  $C_m$ .
- The sensor impedance is dominated by  $G_m$  at low frequency ( $< 10$  kHz) and by  $C_m$  at high frequency ( $> 100$  kHz).
- The electrical capacitance  $C_m$  is function of the oil dielectric properties and is not affected by the oil quality parameters (free acidity and peroxide index).
- The electrical conductance  $G_m$  of the emulsion is function of the oil quality characteristics.

# Working principle of the proposed technique

- In the case of fresh olive oil samples, characterized by a peroxide index  $< 20$ , the emulsion  $G_m$  is function of the olive oil free acidity.



- Samples featuring higher values of free acidity are also characterized by higher values of the emulsion  $G_m$ .
- In the case of oxidized olive oil samples, the presence of non-volatile compounds (such as aldehydes, ketones and hydrocarbons) also contributes to the increase of the emulsion  $G_m$ .

# Design of the portable sensor system



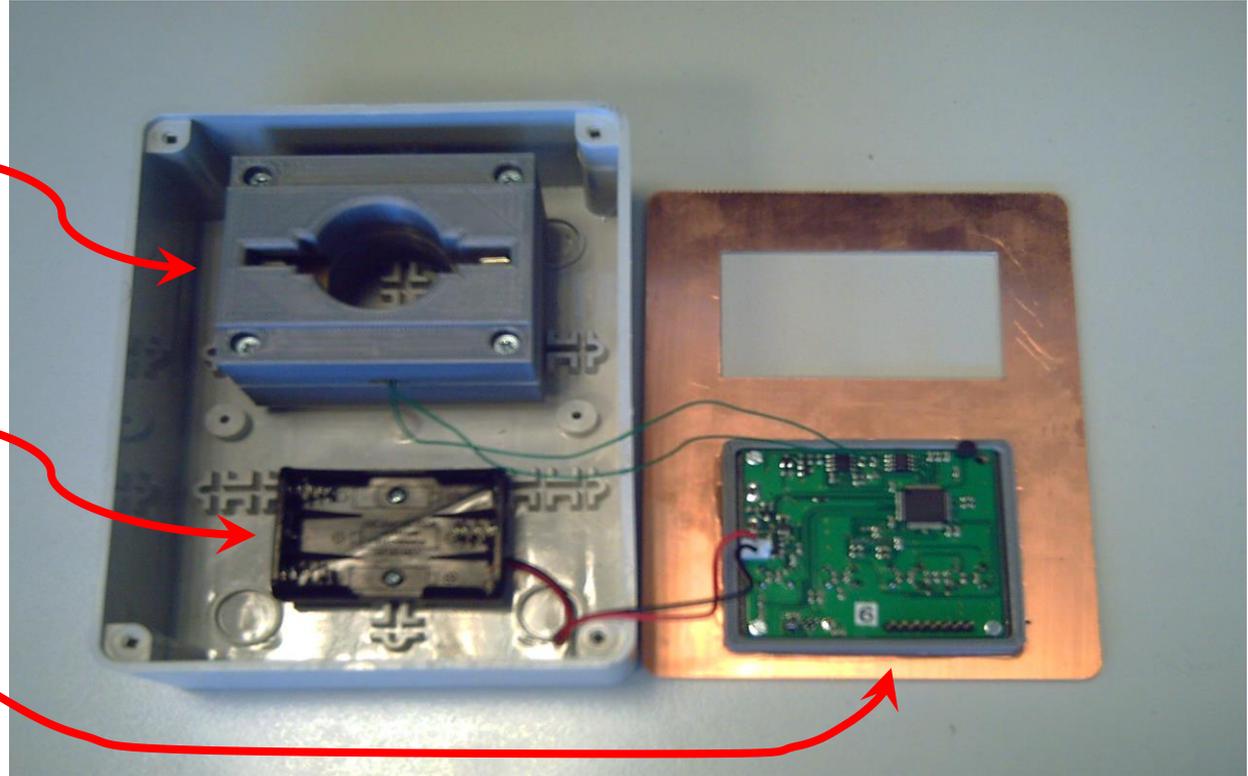
- The portable sensor system has **small size** (11 x 15 x 5 cm), **light weight** (350 g), can be powered by **USB or battery** (3 AAA alkaline batteries) and makes measurements in **about 30 seconds**.

# Design of the portable sensor system

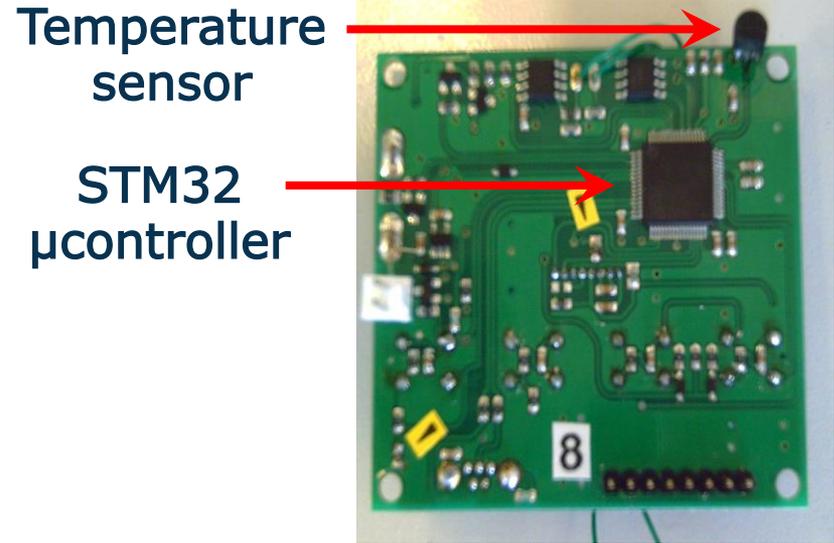
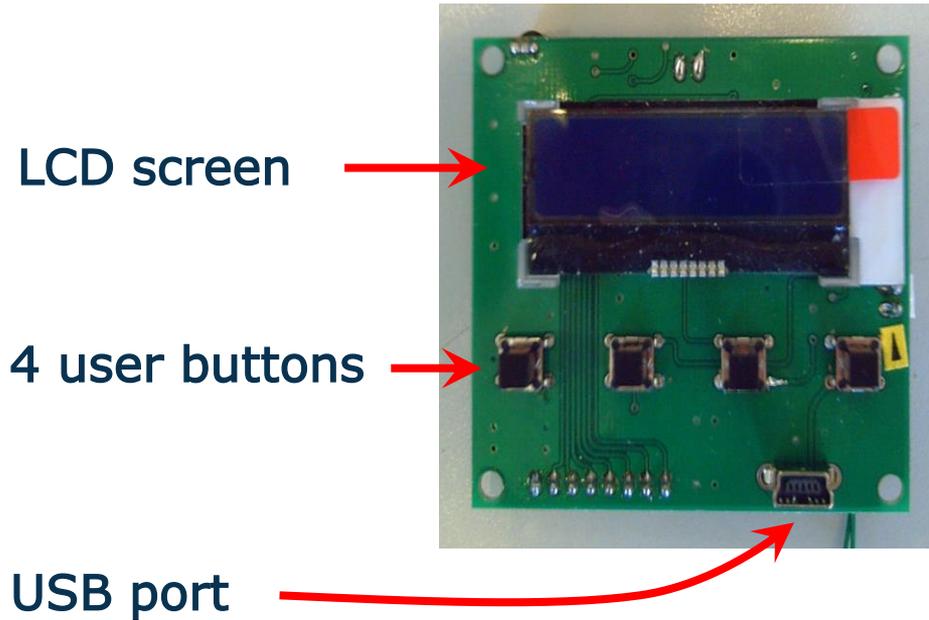
Slot to host the modified Falcon vial

Battery container

Electronic board

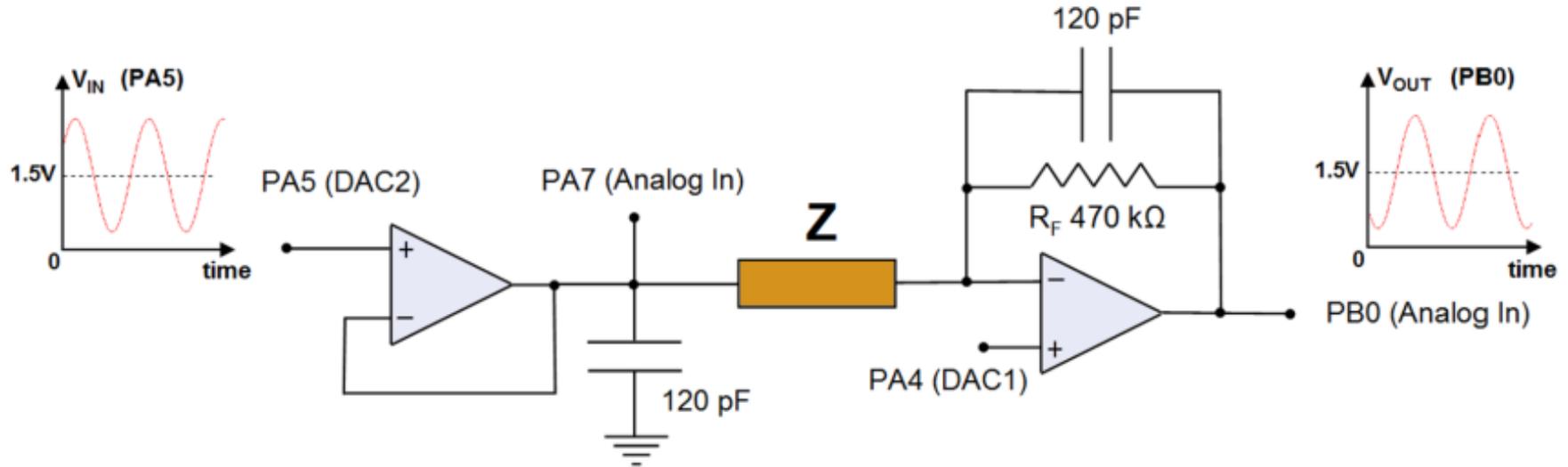


# The electronic board of the sensor system



Ad hoc designed analog circuits for impedance measurement

# The electronic board of the sensor system



# The electronic board of the sensor system

- The sinusoidal voltage signal  $V_{IN}(t)$  (1.5 V offset, 1 V amplitude, 200 Hz) is generated using the microcontroller integrated 12-bit DAC (Digital-to-Analog converter) and applied to the sensor electrodes.
- The sensor current is converted to a voltage  $V_{OUT}(t)$  with a I/V converter.
- The voltage signals  $V_{IN}(t)$  and  $V_{OUT}(t)$  are acquired with the 12-bit ADC (Analog-to-Digital converter) integrated inside the microcontroller.
- The voltage signals  $V_{IN}(t)$  and  $V_{OUT}(t)$  are processed and the sinewave parameters ( $V_{M,IN}$ ,  $V_{M,OUT}$ ,  $\varphi$ ) are calculated.
- The emulsion conductance is calculated as 
$$G_m = \frac{1}{R_F} \times \frac{V_{M,OUT}}{V_{M,IN}} \times \cos(\varphi)$$

# Compensation of the temperature effect

- The portable sensor system has been designed for in-situ measurements in a production environment where the temperature can not be controlled.
- A compensation algorithm has been developed to estimate the emulsion conductance at 23.5 °C ( $G_{m,23.5^{\circ}\text{C}}$ ) from the emulsion conductance at the environmental temperature ( $G_{m,T}$ ) and the temperature value ( $T$ ).
- The compensation algorithm has been implemented with the microcontroller.

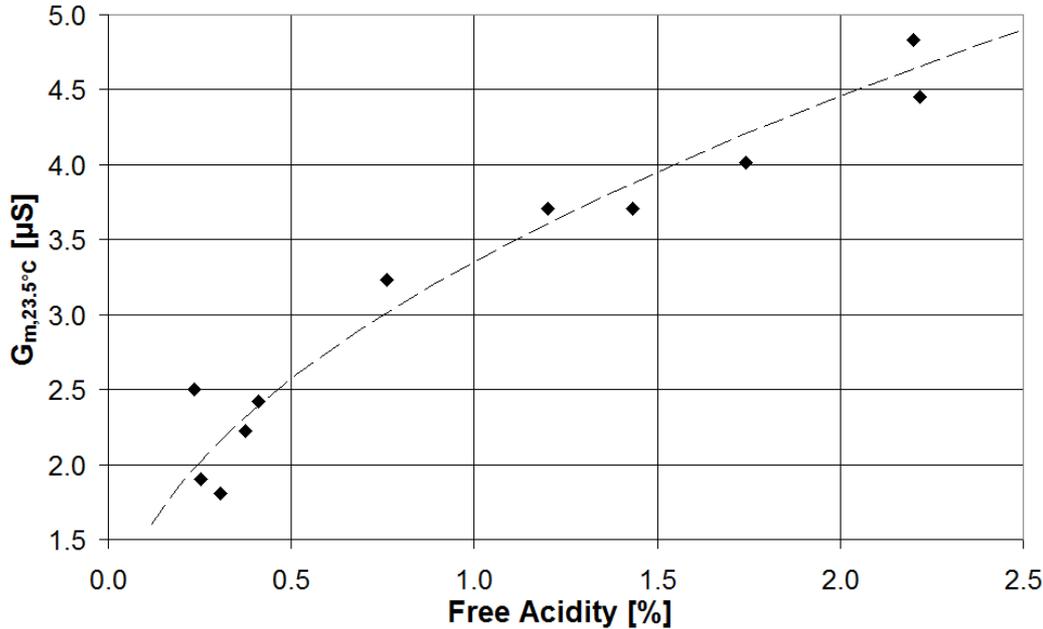
$$G_{m,23.5^{\circ}\text{C}} = \frac{G_{m,T} + 0.0026 \times (T - 23.5)}{1 + 0.0219 \times (T - 23.5)}$$

# Validation of the sensor system

- Tests have been carried out on a set of 17 olive oil samples (6 EVOOs, 3 VOOs and 8 Lampante olive oils).
- A subset of 11 samples (fresh olive oils featuring a peroxide index  $< 20$ ) has been tested and a correlation with the sample free acidity is found.
- In the case of full set of 17 olive oil samples, the emulsion  $G_m$  is affected by both the oil free acidity and the oxidation level, thus a threshold value for  $G_m$  can be set to discriminate EVOOs from lower grade olive oils.

# Tests on fresh olive oil samples

- A correlation exists between  $G_{m,23.5^{\circ}\text{C}}$  and the sample free acidity.

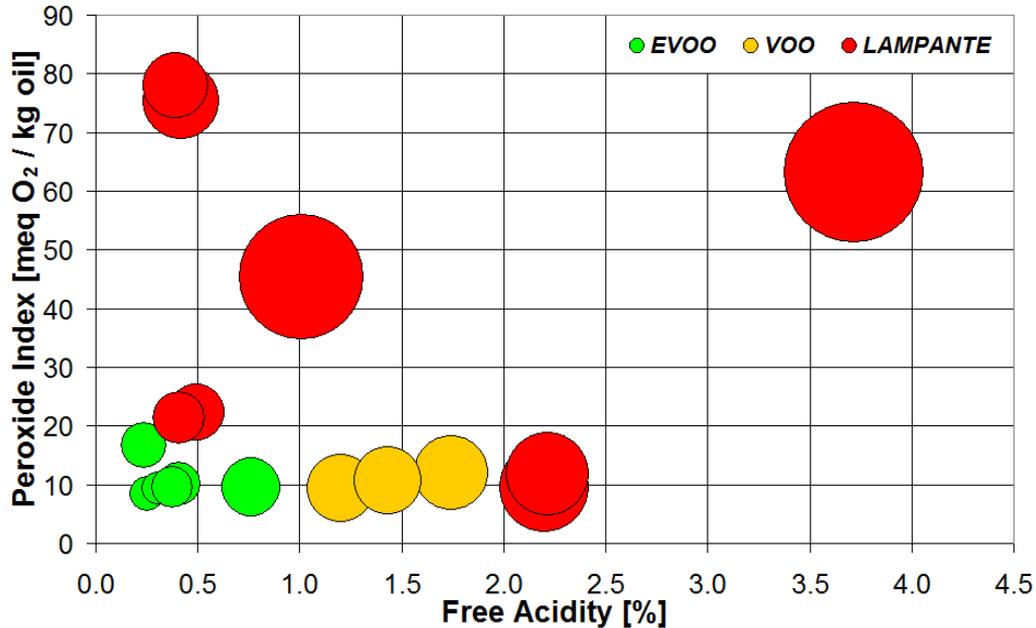


$$FA = \left( \frac{G_{m,23.5^{\circ}\text{C}} - \alpha}{\beta} \right)^2$$

$$\alpha = -0.6856 \quad \beta = 2.6662$$

# Tests on the full set of olive oil samples

- The value of  $G_{m,23.5^{\circ}\text{C}}$  is affected by both free acidity and oxidation level.



- The circle diameter represents  $G_{m,23.5^{\circ}\text{C}}$ .
- EVOO can be discriminated from lower grade oils by setting a conductance threshold  $G_{m,23.5^{\circ}\text{C,TH}} = 2.7 \mu\text{S}$ .
- $G_{m,23.5^{\circ}\text{C}} < G_{m,23.5^{\circ}\text{C,TH}} \rightarrow \text{EVOO}$

# References

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# Thanks for your attention