Analytical Solutions Addressing
Olive Oil Quality Issues

Diego Luis García González

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Standard Method (IOC/EU) of Sensory Assessment
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- **COI/T.20/DOC. 4/REV.1 - 2007** Sensory analysis: general basic vocabulary
- **COI/T.20/DOC. 5/REV.1 - 2007** Glass for oil tasting
- **COI/T.20/DOC.14/2013** - Guide for the selection, training and monitoring of skilled virgin olive oil tasters
- **COI/T.20/DOC.15/Rev. 7 - 2015** - Sensory analysis of olive oil - method for the organoleptic assessment of virgin olive oil (February 2015)

Quality Improvement

80s

2017
Standard Method (IOC/EU) of Sensory Assessment

**IMPROVEMENTS**

- **COI/T.20/DOC. 4/REV.1 - 2007** Sensory analysis: general basic vocabulary
- **COI/T.20/DOC. 5/REV.1 - 2007** Glass for oil tasting
- **COI/T.20/DOC.14/2013** Guide for the selection, training and monitoring of skilled virgin olive oil tasters
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Panelist training

Reference Materials

Statistics

Accreditation and Harmonization

A standardized instrumental method to support panel test work
Shelf life and freshness control

Shelf-Life
Quality decay
Needs of analytical support
Health claim and phenol analysis

http://ec.europa.eu/nuhclaims/?event=search#

"Polyphenols in olive and olive oil"

Protection of LDL particles from oxidative damage

"containing at least 5 mg of hydroxytyrosol and its derivatives (e.g. oleuropein complex and tyrosol) per 20 g of olive oil"

How to "translate" this in analytical terms (Analytical method)

"Only 11% of submitted health claims are authorized"
Objectives

Panel test improvement (quantitative panel test)

Volatile analysis to support panel tests

Analytical tolos for Shelf life control

Health claim and
**Standard Method (IOC/EU) of Sensory Assessment**

**IMPROVEMENTS**

- COI/T.20/DOC. 4/REV.1 - 2007 Sensory analysis: general basic vocabulary
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**Reference Materials**

- Selection of volatile markers
- Chemical characterization of sensory defects

**Natural (VOO)**

- Highly representative
- Not homogeneous year after year
- Insufficient amount for a continuous monitoring

**Synthetic (volatile compounds + estable matrix)**

**CHARACTERIZATION BY VOLATILE COMPOUNDS**
Sampling strategy

6 Sensory panels

- P1: 36 samples
- P2: 32 samples
- P3: 14 samples
- P4: 41 samples
- P5: 30 samples
- P6: 27 samples

180 samples → 60 samples

- 12 EVOO
- 30 VOO
- 18 LVOO

26 Fusty
11 Rancid
6 Musty
4 Winey-vinegary
1 Frostbitten
2 Others (brine)

Analytical techniques:

- SPME-GC-FID
- SPME-GC-MS
- TDU-GC-MS
- GC-Olfactometry
- FGC-E-Nose
- Distillation-NMR
UNIFIED PROTOCOL FOR SAMPLING, SHIPPING AND STORING
Participants.

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Median of defect

DO WE STUDY ALL THE RANGE? YES

[Graph showing the median of defect (Md) for different types of oil, with VOO, EVOO, and LVOO indicated.]
**Factor 1: 62.16%**

- **Md=6.2 (Fusty)** Excessive concentration of ethanol
- **MD=5.7 (Winey-vinegary)** Excessive concentration of ethanol
- **Md=9 (Rancid)** Excessive concentration of Hexanal

**Factor 2: 10.12%**
Median of defect (Md) (0-10)

Outliers

-2
0
2
4
6
8
10

VOO
EVOO
LVVOO
Analysis with all volatile compounds (82)

All variables (82)

Active

EV

L

EV

V

V

V

VV

L

EV

EV

EV

V

V

V

V

L

L

VV

L

EV

EV

V

V

L

L

V

V

EV

EV

L

EV

EV

EV

V

L

L

L

Noise + Informative data
All volatile compounds (82)

Selection by ANOVA+Brown-Forsythe (p<0.05)

- EVOO vs. the rest
- EVOO vs. VOO
- VOO vs. LVOO
- EVOO vs. LVOO

<table>
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<th>Octane</th>
<th>Pentane</th>
<th>Pentan-3-ol</th>
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<tbody>
<tr>
<td>2-Methyl-1-propanol</td>
<td>3-Methyl-1-butanol</td>
<td>Isobutiric acid</td>
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<td>Sensory defect</td>
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<td>Intense</td>
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<td>Bottled</td>
<td>2,6-Nonadienal</td>
<td>0.09</td>
<td>Cucumber</td>
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Checking with previous works


Morales et al. (2005). Food Chem. 91, 293-301.
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**Breakdown analysis between designations**

**Box & Whisker plots**
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**Box & Whisker plots**
Samples with *winey-vinegary* defect

**EURO28**  
*Md*=3

**UNIBO7**  
*Md*=2.3

Main defect, agreement in panel tests

Other samples where *winey-vinegary* has been mentioned:

**UP6, ITERG1, EURO26, EURO34, EURO21, UZZK6**

**Ethyl acetate**

**Ethanol**

**Acetic acid**

**Alcoholic fermentation**

**Acetobacteria**

Ethanol  →  Acetaldehyde  →  Acetic acid

A-DH  =  Alcohol dehydrogenase
AL-DH  =  Aldehyde dehydrogenase

Cytoplasm
Acetic acid (mg/kg)

Vinegary defect detected by panellists

Odour Threshold = 0.5 mg/kg
Winey-vinegary defect: ongoing work

Chemical characterization of defects

Selection of markers

Design of formulations

ISO Characteristics

Representative? Yes

Stable? No

Homogeneous? No

WP3 - Analytical solutions addressing olive oil quality issues
Supporting documents for Workpackage 3 - Issues related to the sampling and analytical activities in WP3
Olive Oil Quality Assurance

Document prepared by CSM

OLEUMPROJECT.EU | #OLEUM2020
Concluding remarks

- Developing RMs can be applied for training panels, continuous evaluation of panellists, and harmonization activities without the problem of having limited quantities.

- The analysis of volatile can support the work of a panel test in different aspects, overall when doubts between categories (e.g. EVOO-VOO).

- Adressing VOO quality from different perspectives (volatiles, shelf life, phenols) allows incorporating recent analytical advances into quality control.
Thank you for your attention

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Instituto de la Grasa (CSIC)
Needs for a Reference Materials

Natural (VOO)
- Highly representative
- Non homogeneous between years
- Insufficient amount for a continuous monitoring

Emulated with volatile compounds + stable matrix
- Aroma that resembles this of sensory defects.
- Homogeneous year after year (closer to ISO definition of RM).
- Sufficient amount for a continuous monitoring.

Characterization of Volatile Compounds
- Selection of volatile markers
- Chemical characterization of sensory defects
Objectives

- Calibration of Sensory Panels
- REVISION of “Guide for the selection….” COI/T.20/DOC.14/2013

Volatile compounds

- Method to detect/quantify volatile compounds
- Freshness assessment
- Guarantee Health Claim
- Better reliability
- Hydroxytyrosol and derivatives
- Method + validated software

Non volatile volatile compounds

Develop/Validate

RM

Guarantee Health Claim

Better reliability

Hydroxytyrosol and derivatives
Strategy

Formulations

Statistical Sensory Wheel/Update of literature
List of descriptors for volatile compounds
Previous experiences

Open debate (free-choice sensory description)

GC analysis

Adjustment of concentration balance