Towards the preservation of olive mill leaf quality with an innovative drying technique

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Olea europaea L. (Oleaceae)

- Evergreen tree
- Native in the Mediterranean area
- Worldwide cultivation
- Olive oil and table olive production
- Bioactive compounds in tissues (phenolic compounds, terpenoids etc.)

<u>Olive leaves</u>

- Traditional medicine
- Food supplements, herbal preparations

Tsimidou & Papoti (2010), Academic Press, 349

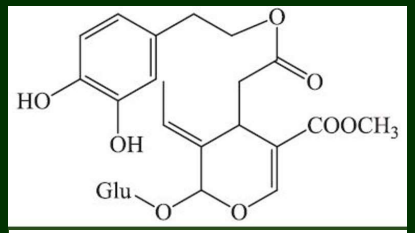
Olive leaves

Bioactive compounds

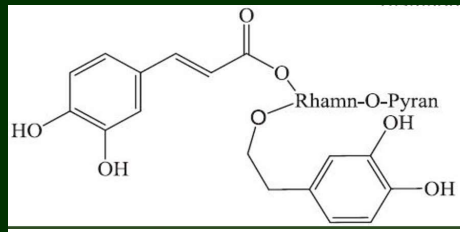
- Phenolic compounds
  - Oleuropein (≤ 143.2 g/kg dw)
  - Verbascoside (≤ 18.6 g/kg dw)
  - Flavonoids
    - Luteolin-7-O-glucoside (≤ 6 g/kg dw)
    - Apigenin-7-O-glucoside (≤ 2.33 g/kg dw)
- Terpenoids
  - Oleanolic acid (≤ 34.5 g/kg dw)
  - Maslinic acid (≤ 7.3 g/kg dw)

Health-promoting activities

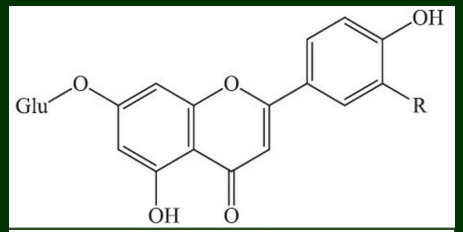
- Antioxidant
- Antimicrobial
- Cardioprotective
- Antidiabetic



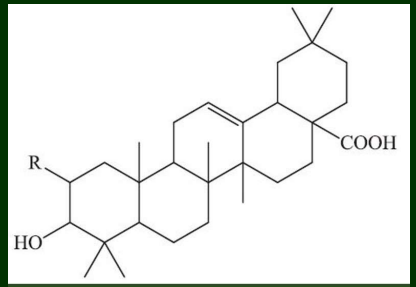
Oleuropein



Verbascoside



R = OH: Luteolin-7-O-glucoside
R = H: Apigenin-7-O-glucoside



R = OH: Maslinic acid
R = H: Oleanolic acid

Nenadis et al. (2021), Academic Press, 65; Özcan & Matthäus (2017), Eur Food Res Technol, 243

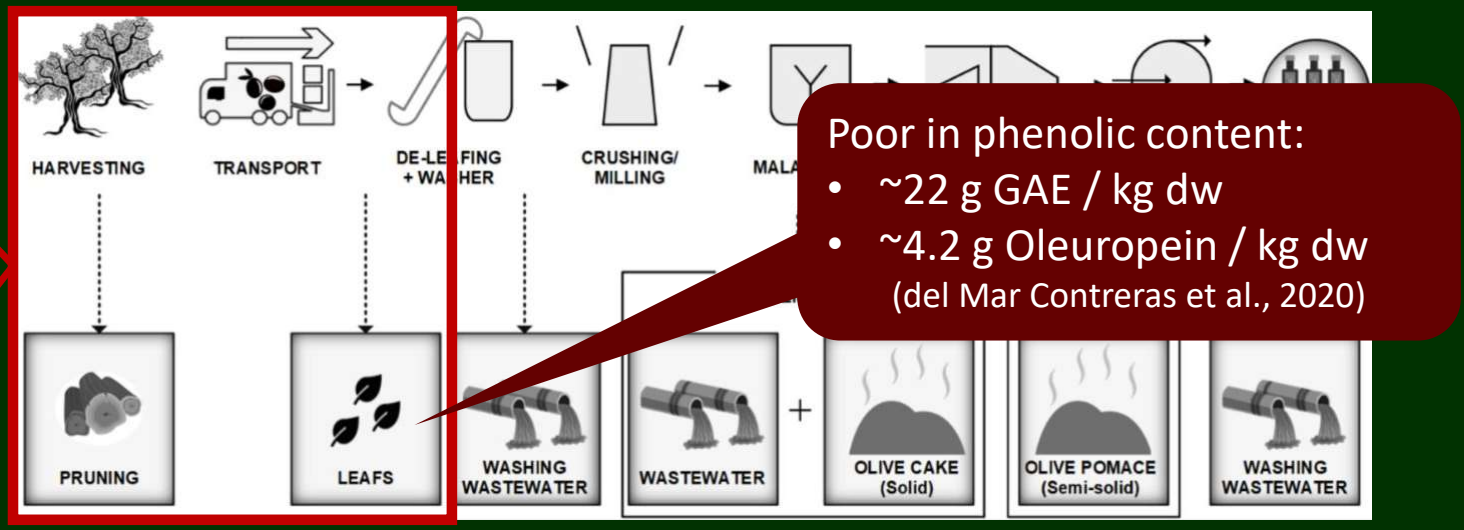
Olive leaves

Availability

- Throughout the year
- Periodically, as byproduct of olive cultivation
  - Tree pruning (~25 kg per tree, including branches)
  - Olive fruit harvest (4 – 10 % of the weight of the drupe load delivered to the mill for processing)

Fertilizer

Pharmaceutical, Food and Feed



Poor in phenolic content:
- ~22 g GAE / kg dw
- ~4.2 g Oleuropein / kg dw
  (del Mar Contreras et al., 2020)

Cagliari et al. (2022), Brazilian J. Food Technol., 25; Ribeiro (2021), PhD Thesis, Porto; del Mar Contreras et al. (2020), Food Chem., 314

Olive leaves

Factors influencing the composition

- Natural variation
  - Biotic (cultivar, age of trees and leaves etc.)
  - Abiotic (pedoclimatic conditions, agricultural practices etc.)
- Postharvest practices
  - Storage conditions
  - Drying technique

- First process
- Deactivation of oxidative enzymes
- There is no commonly accepted technique or conditions

Common techniques

- Air – drying (≥ 2 days, uncontrollable conditions)
- Freeze – drying (≥ 1 day, high cost)
- Oven – drying (40 – 120 ^{o}C)

Emerging techniques

- Microwave – drying
- Infrared – drying

- Eco-friendly
- Fast
- Effective

Within the frame of the research project OliveFeed, aiming to exploit by products from olive mills as raw materials for the production of extracts to be incorporated in broiler's feed…

National and Kapodistrian University of Athens

University of Thessaly

Aristotle University of Thessaloniki

Olive Feed

Union of Agricultural Cooperative of Lakonia (EASL)

Viozokat S.A.

AVMAP GIS S.A.

Task: examine the quality of leaves produced from olive leaves, located in Lakonia, the main olive oil producing region in Greece

European Union
European Regional Development Fund

HELLENIC REPUBLIC
MINISTRY OF DEVELOPMENT AND INVESTMENTS
SPECIAL SECRETARIAT FOR ERDF & CF PROGRAMMES
MANAGING AUTHORITY OF EPAnEK

EPAnEK 2014-2020
OPERATIONAL PROGRAMME
COMPETITIVENESS
ENTREPRENEURSHIP
INNOVATION

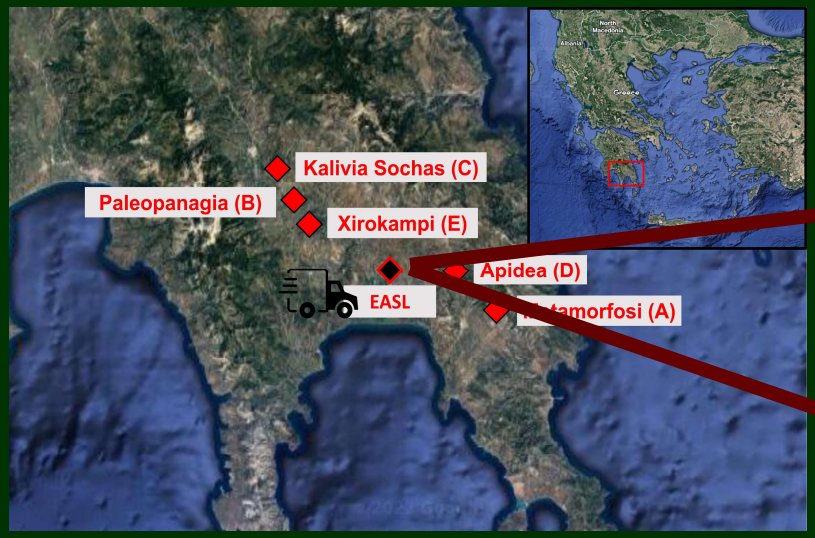
ΕΣΠΑ 2014-2020
ανάπτυξη - εργασία - αλληλεγγύη
Partnership Agreement 2014 - 2020

Co-financed by Greece and the European Union

... we received various dried olive leaf samples from the region of Lakonia...

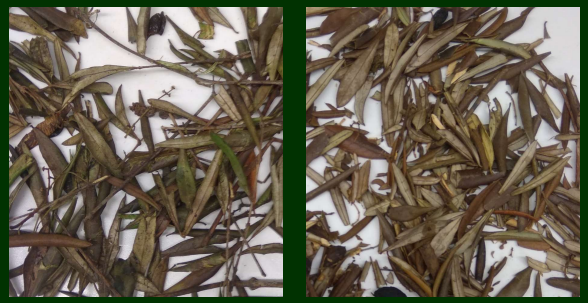
- 31 samples (1-31), cv Koroneiki
- Collection from five olive mills (A – E)
- Transportation to Union of Agricultural Cooperative of Lakonia (EASL)
- Drying: immediately after arriving to EASL, conventional oven with trays, set at 70 and 140 °C

- Transportation to
  - Aristotle University of Thessaloniki (AUTh)
  - National and Kapodistrian University of Athens (UOA)



...and examined their appearance, their moisture content...

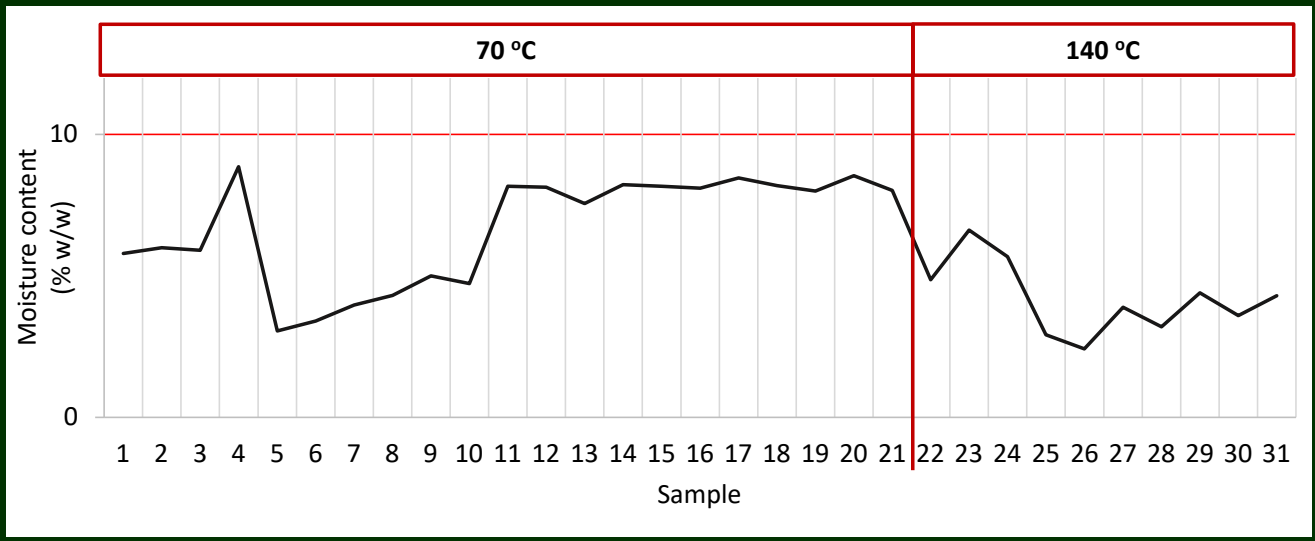
Sample inspection



70 °C 140 °C

- Burned-like odor
- Loss of green color
- Soil residues, woody parts, fruit peels

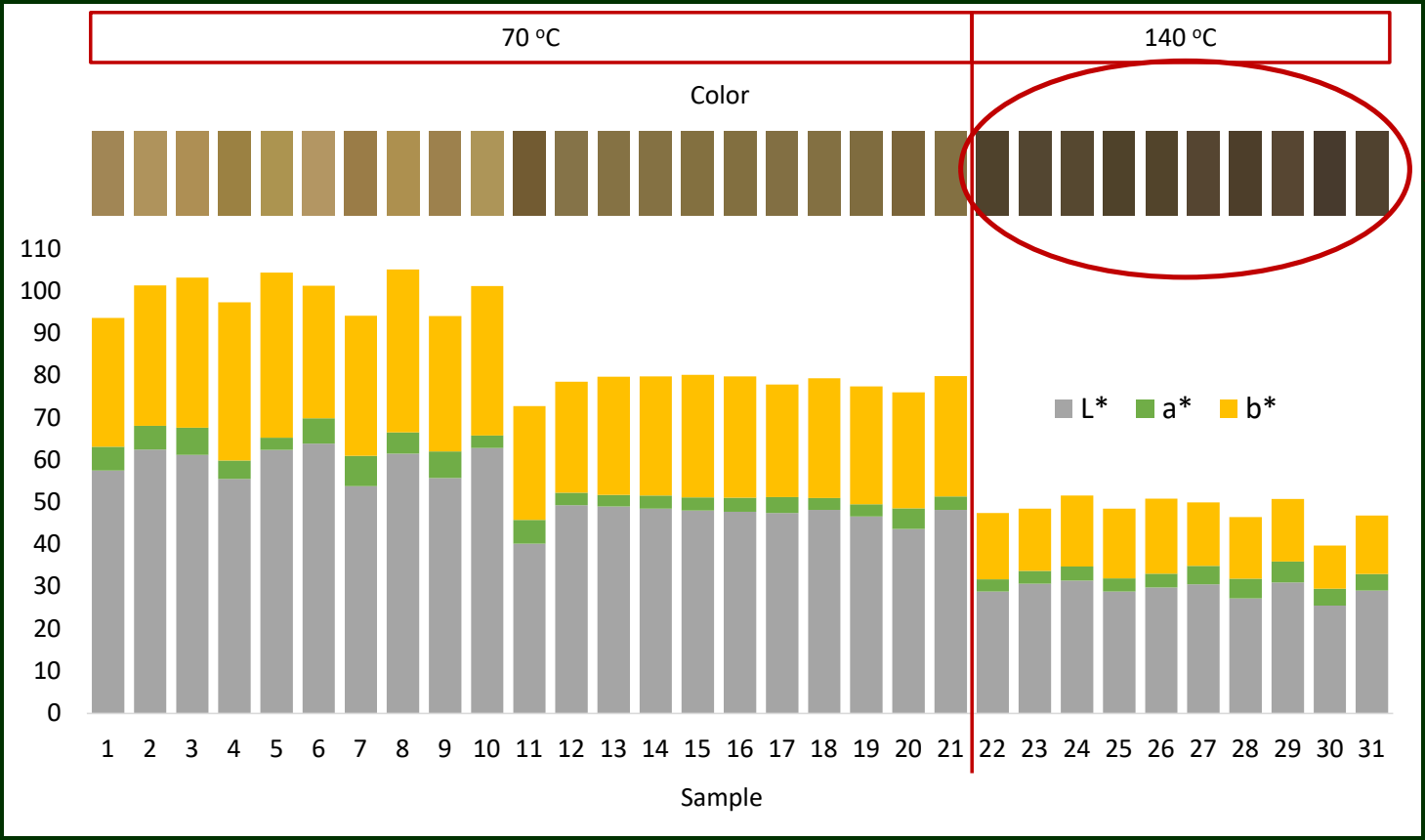
Moisture content (Moisture analyzer DAB 100–3)



- Effective drying
- Residual moisture < 10 % w/w: adequate preservation according to European Pharmacopoeia

...their color...

Color (CIE L*a*b*)



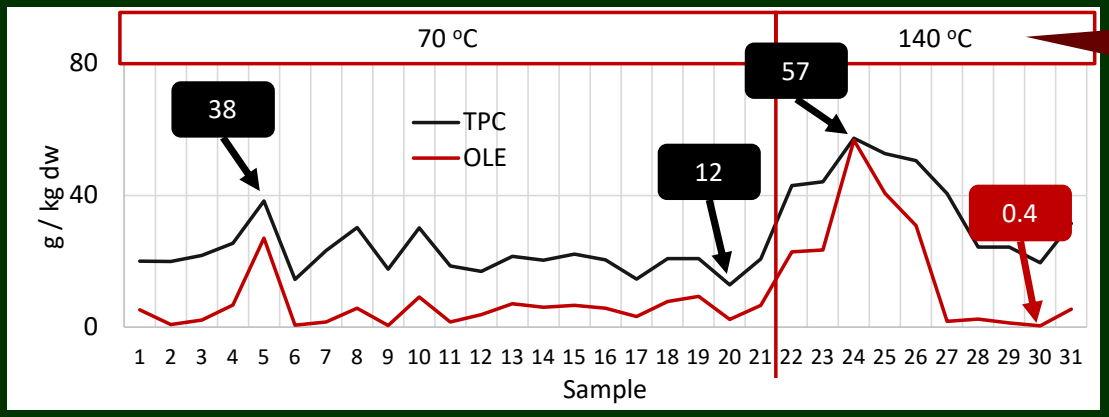
- Maillard reactions
- Caramelization
- Chlorophyll degradation

(Boukhiar et al., 2022)

Inefficient
post-harvest treatment
or
drying process

...and their phenolic content

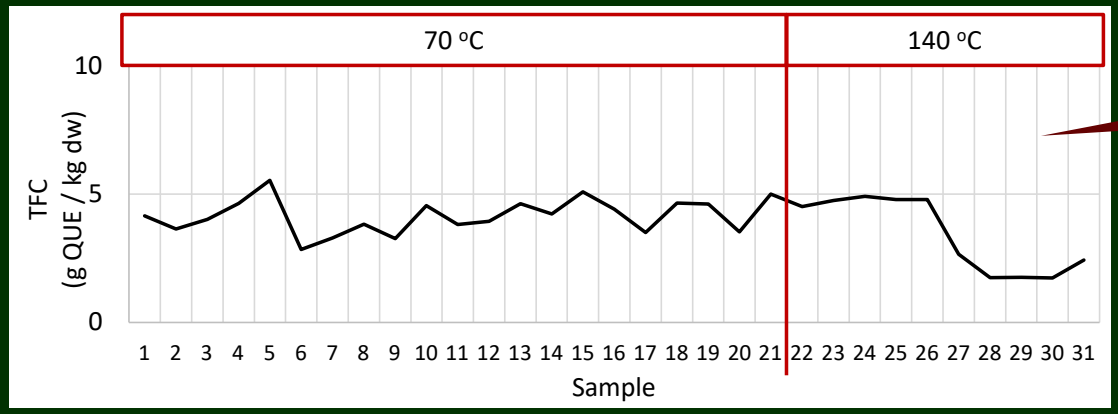
Total Phenolic Content (TPC, Folin-Ciocalteu assay, gallic acid equivalents, GAE)
Oleuropein content (OLE, HPLC, Savournin, 2001, JAFC, 49)



Drying at temperatures > 100 °C for short time
- Higher TPC (Ahmad-Qasem et al., 2013)
- Phenoloxidase deactivation (Ortega-Garcia et al., 2008)
  - high temperature
  - reduced water activity

No clear trend regarding temperature was evidenced

Total Flavonoid Content (TFC, $AlCl_3$ method, Cvek et al., 2007, Quercetin equivalents, QUE)



TFC: 11 – 22 g/kg dw
(Papoti et al., 2018)

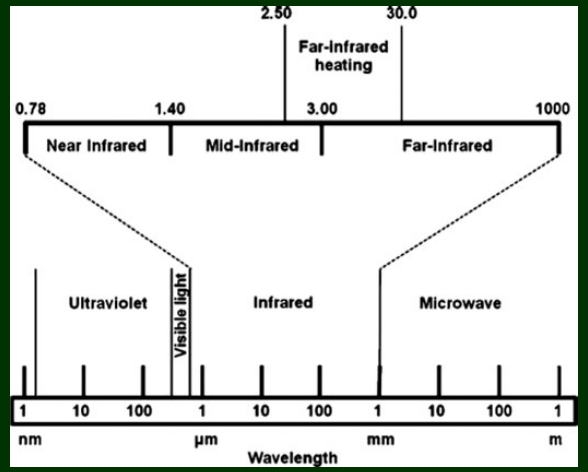
Olive leaves collected from mills during olive oil production are rather of poor quality

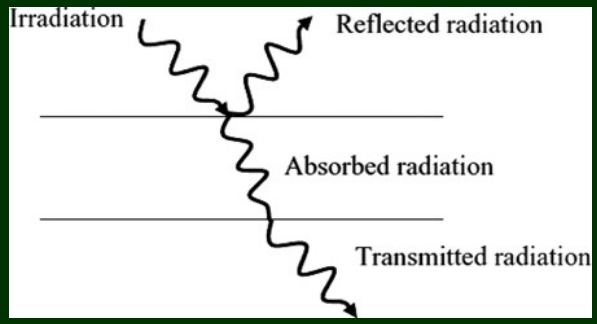
Ahmad-Qasem et al. (2013), Food Res. Int., 50; Ortega-Garcia et al. (2008), Tree Physiol., 28; Papoti et al. (2018), Foods, 7

As various factors may be responsible for such findings, and trying **to contribute** towards a faster handling of olive leaves regarding drying and quality preservation, the present work provides evidence for the efficiency of infrared drying

Infrared radiation (IR)

- 3 Categories
- Reflection, absorption, transmission
- Suitable for drying food stuff (water absorption bands: 3, 4.7, 6 and 15.3 μm)
- IR energy is absorbed in a narrow region close to the surface of the material
- Penetration depth (Hashimoto and Kameoka, 1997)
  - FIR: 0.21 – 0.64 mm → suitable for drying thin layers
  - NIR: 0.38 – 2.54 mm → suitable for drying thicker materials
- Direct and uniform heating of sample without interfering with air, no preheating
- Intermittent emission: tempering period → moisture and heat diffusion → shorter drying time → limitation of bioactive degradation
- Scaled-up for industrial use: trays or conveyor belts





Sakare et al. (2020), Food Eng. Rev., 12; Riadh et al. (2015), Dry. Technol. 33

Intermittent Near Infrared Drying (NIRD) of olive leaves

Toshiba TL1-AC25CZA(BS) digital toaster oven

- 2 NIR elements
- auto-adjusted intermittency

Optimization

- Central composite design
- Independent variables
  - Temperature: 71 – 116 ^{o}C
  - Time: 3 – 23 min

Optimum conditions: 116 ^{o}C, 3 min

Best preservation of color and TPC

Intermittent Near Infrared Drying (NIRD) of olive leaves

- cv Koroneiki
- Harvested from American Farm School in Thessaloniki
- Transportation to Aristotle University of Thessaloniki (AUTh)
- Washing

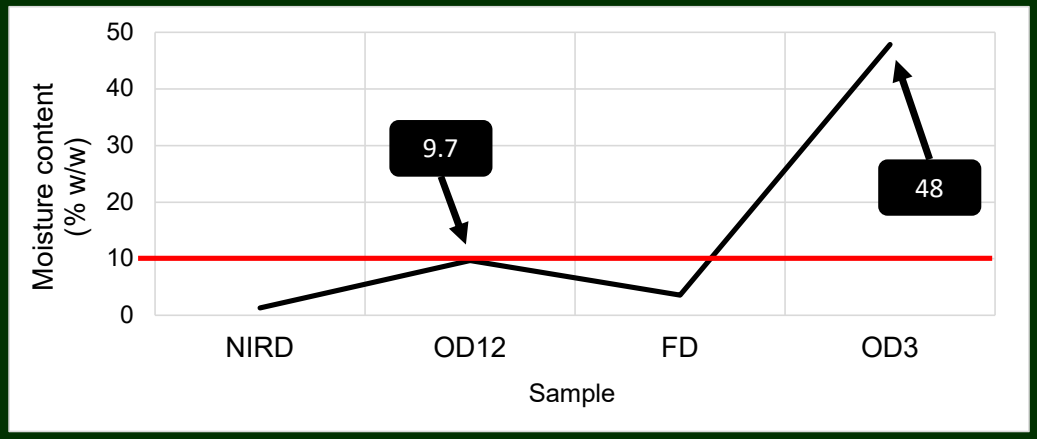
Drying

- Near Infrared oven (NIRD): 116 °C, 3 min — Optimum conditions
- Conventional oven: 120 °C — Most common technique; Used by EASL
  - Until constant weight; 12 min (OD12)
  - Comparison with NIRD; 3 min (OD3)
- Freeze dryer (FD): 24 h — Non-thermal; Used for food preservation; Expensive

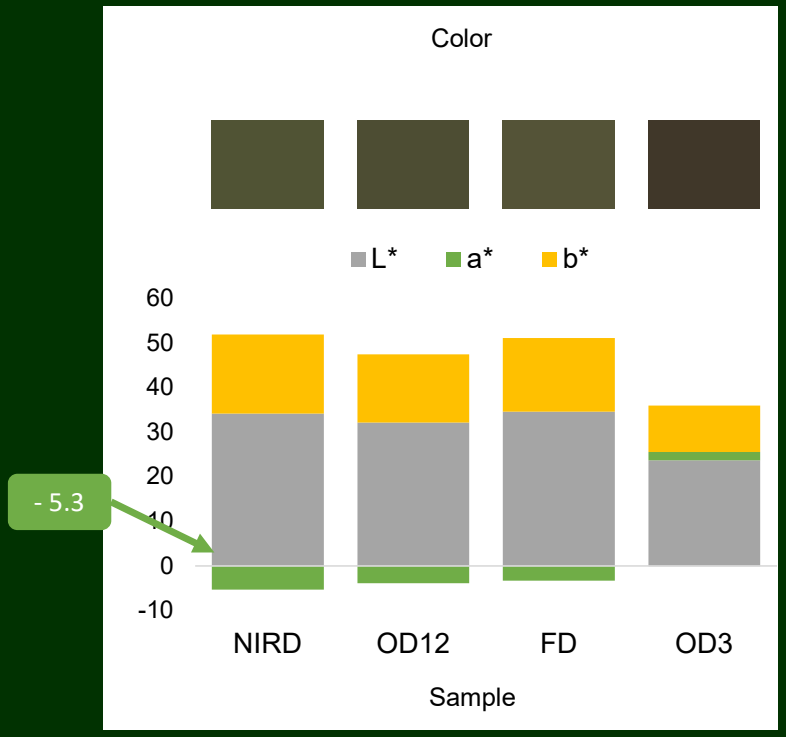
Intermittent Near Infrared Drying (NIRD) of olive leaves

Sample inspection

Moisture content (Moisture analyzer DAB 100–3)



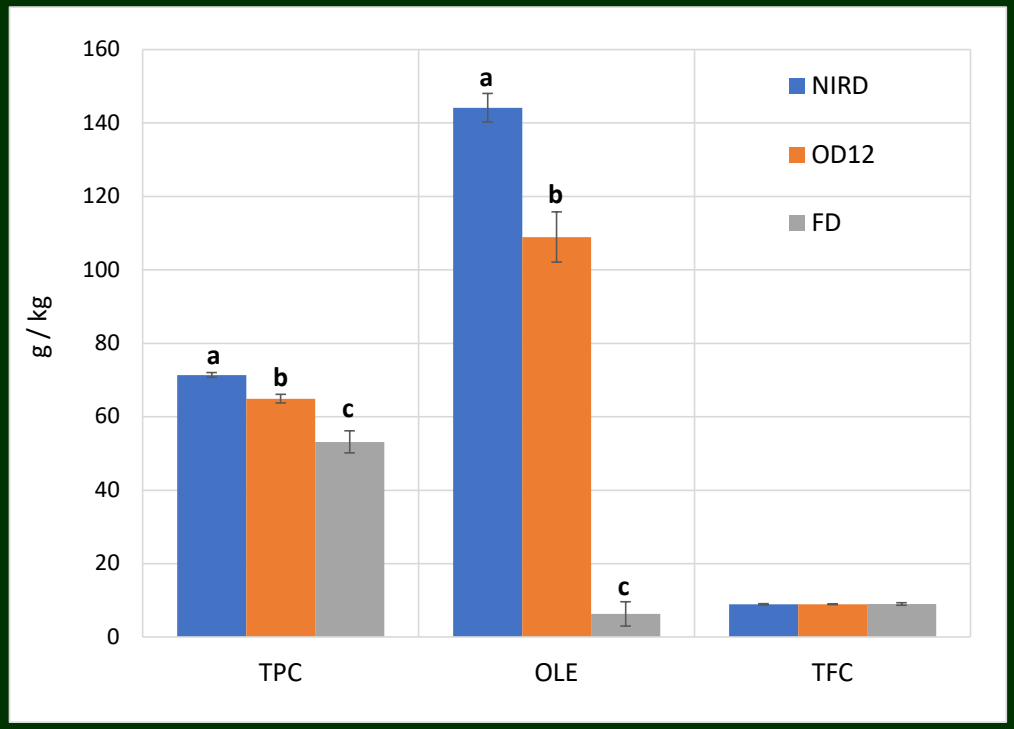
Color (CIE L*a*b*)



OD3 excluded from further analysis

Intermittent Near Infrared Drying (NIRD) of olive leaves

Total Phenolic Content (TPC, Folin-Ciocalteu assay, gallic acid equivalents, GAE)
Oleuropein content (OLE, HPLC, Savournin, 2001, JAFC, 49)
Total Flavonoid Content (TFC, AlCl3 method, Cvek et al., 2007, Quercetin equivalents, QUE)



Near Infrared Drying
- most efficient technique
- preservation of TPC and OLE

Freeze-drying
- Does not guarantee the maximum OLE content

All techniques were efficient for the preservation of TFC

Conclusions - Future directions

- Olive leaves, especially when collected as byproducts from olive mills, should be handled appropriately to be used as a raw material for further high-added value applications in Pharmaceutical, Food and Feed sectors
- Postharvest strategies, such as storage and drying, should be carefully chosen for the preservation of the bioactive compounds of olive leaves
- Drying olive leaves at a high temperature for a short time provides the highest content of phenolic compounds
- Near Infrared radiation is an effective, quick and innovative means of drying, which can be scaled-up for industrial use

Acknowledgements

We would like to thank the Union of Agricultural Cooperative of Lakonia (EASL) for collecting the samples, their handling and shipment for analyses.

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Thank you for your attention!