

Advancing headspace extraction workflows for comprehensive VOC profiling of virgin olive oil using GC×GC–MS

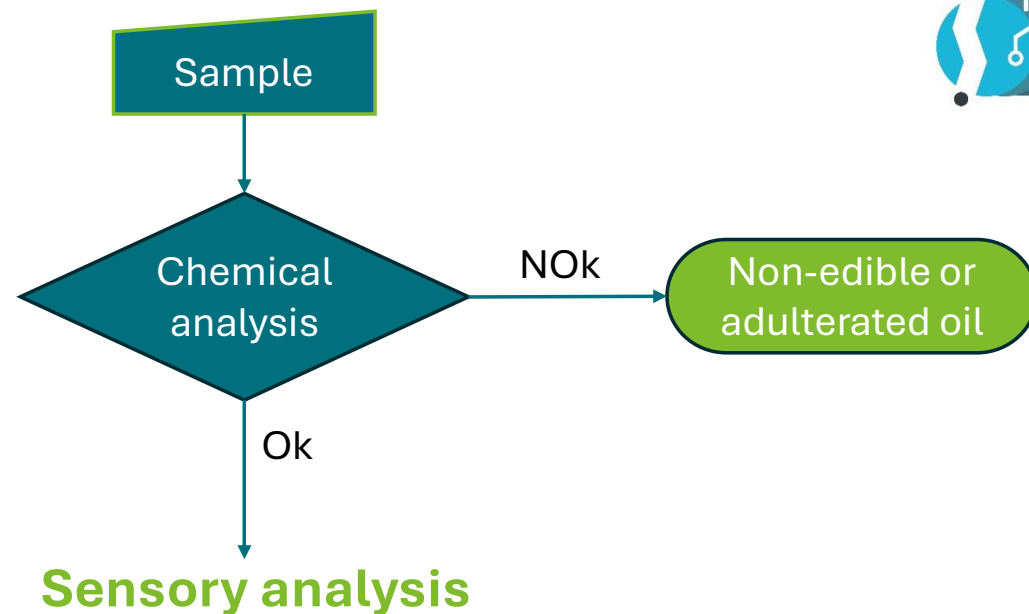
Irene Digiglio, Damien Eggermont, Natasha Damiana Spadafora, Tommaso Ugolini, Lorenzo Cecchi, Nadia Mulinacci, Giorgia Purcaro

*19th international symposium on hyphenated techniques in
chromatography and separation technology*



Olive oil (OO)

- Commercial quality
 - Extra Virgin (EV) OO
 - Virgin (V) OO
 - Lampante (L) OO
- Assessment
 - Chemical analysis
 - Sensory analysis
 - “Defects”
 - “Fruitiness”



Median	EVOO	VOO	LOO
Defects	0	≤ 3.5	> 3.5
Fruitiness	> 0	> 0	





Olive oil – sensory analysis



- Sensory analysis
 - Human panellist
 - Limited capacity
 - Time-consuming and costly
 - Regional differences and misalignment
 - Subjectivity
 - Training issue

Sensory analysis

Median	EVOO	VOO	LOO
Defects	0	≤ 3.5	> 3.5
Fruitiness	> 0	> 0	

Alternative/helping analytical techniques ?





Olive oil – sensory analysis



- Sensory analysis
 - Based on (semi-)Volatile Organic Compounds (sVOCs)
 - “Fruitiness” → Lipoxygenase (LOX) pathway C₅-C₇ metabolites
 - Ex: 1-penten-3-one, 1-penten-3-ol, (E)-hex-2-enal, ...
 - “Defects” → microbial processes, oxidative degradation, etc. C₇-C₁₁ metabolites
 - Ex: saturated and monounsaturated aldehydes, dienals, branched aldehydes, carboxylic acids, and ketones

Alternative/helping analytical techniques ?





Olive oil – Analytical techniques



- sVOCs analysis
 - SAFE
 - Gold standard
 - Long
 - Tedious
 - Not green



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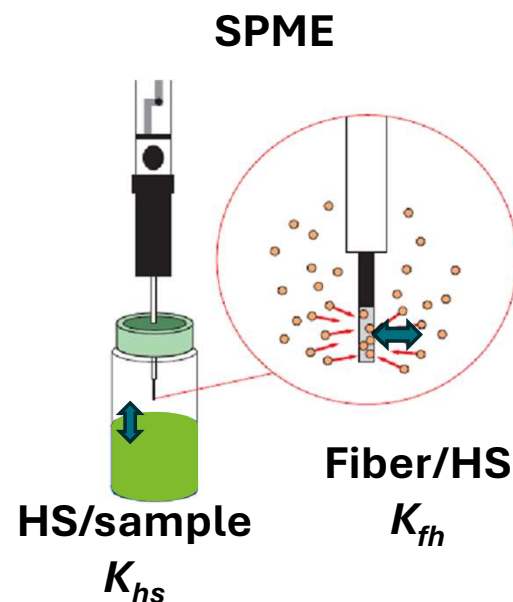




Olive oil – Analytical techniques



- sVOCs analysis
 - SAFE
- Microextraction techniques
 - Many different geometries/configurations
 - Headspace solid-phase micro-extraction
 - Automation capability
 - Solvent free
 - Lower sample uses
 - Easiness
 - Green and practical



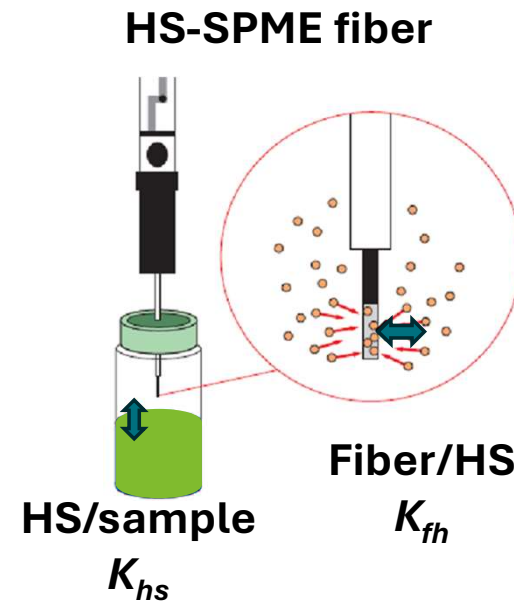
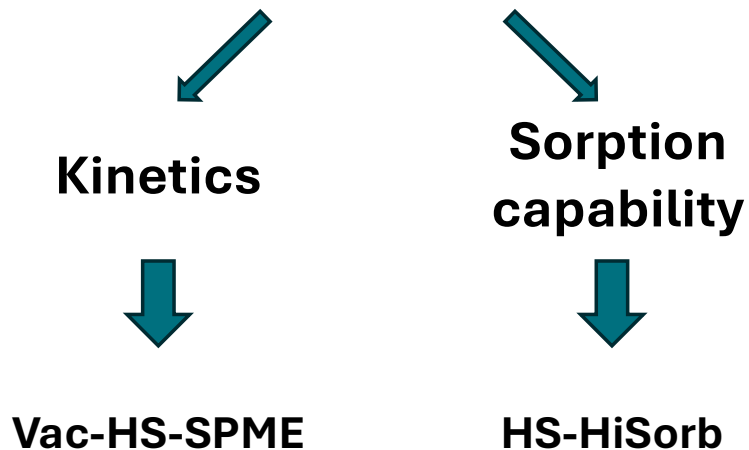
- Fiber
- Arrow
- HiSorb
- Stir-Bar
- Thin-Film
- In-tube
- Blade
- ...





Olive oil – Analytical techniques

- SPME different configurations/geometries
 - HS-SPME fiber
 - Sensitivity sometime limited
 - Slow equilibrium

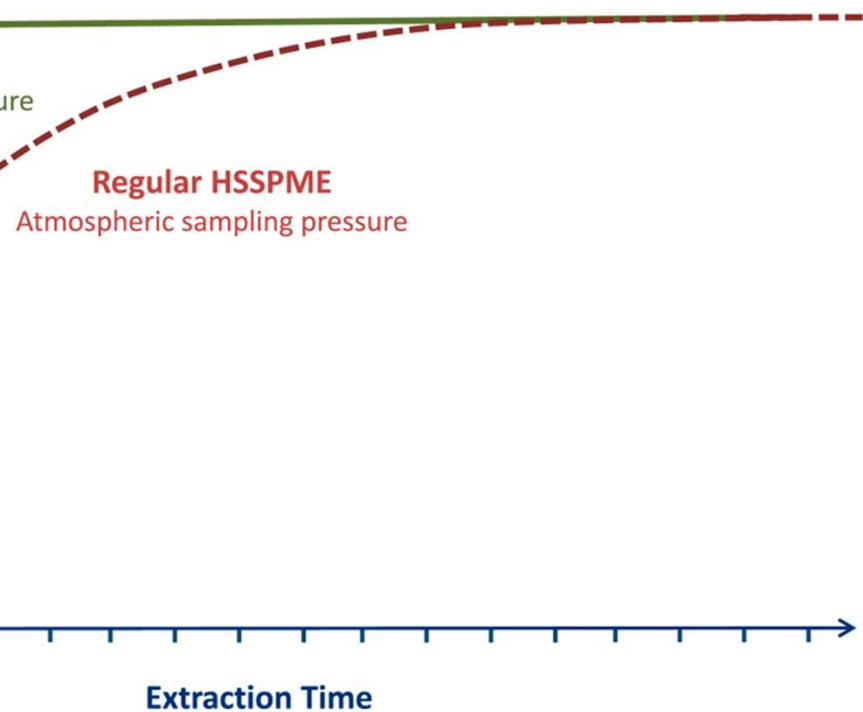




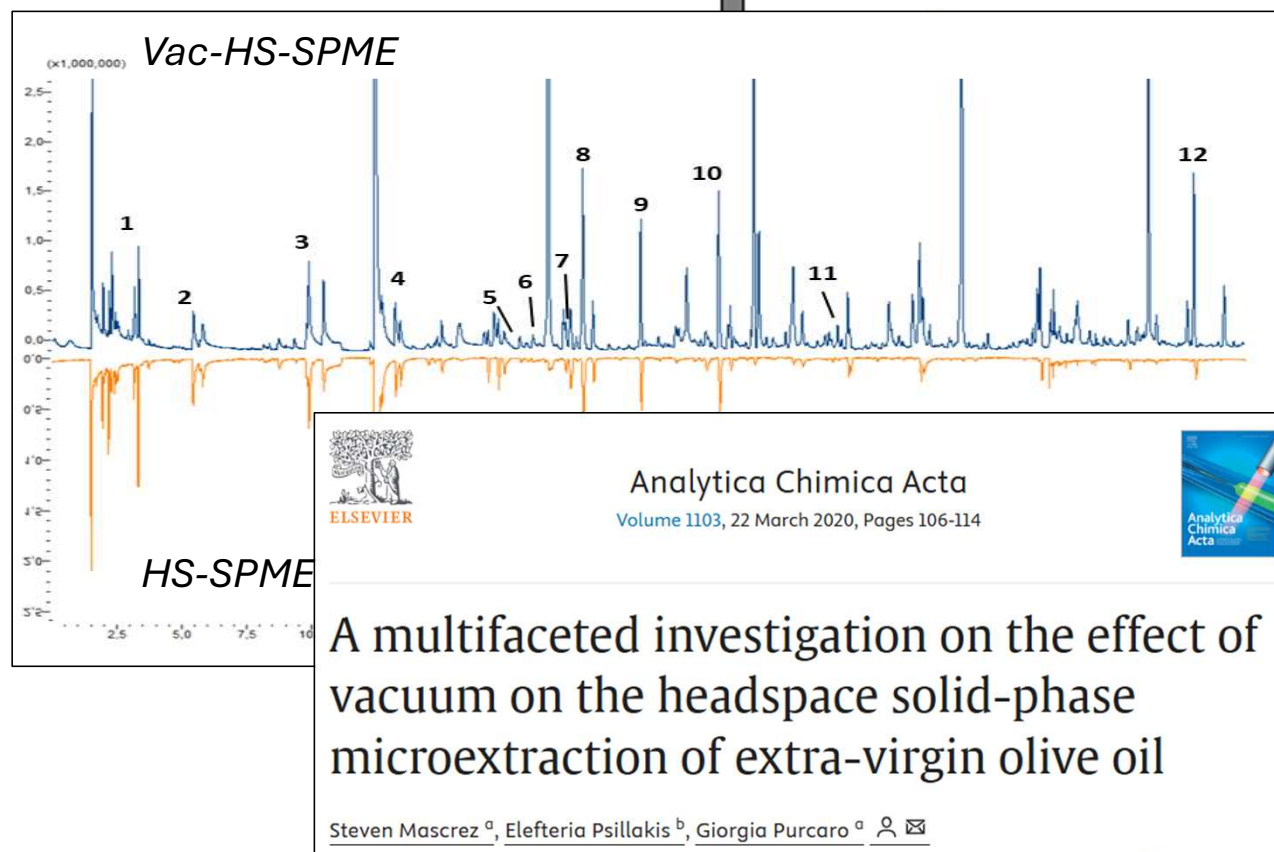
Olive oil – Analytical techniques



- SPME different configurations/geometries
 - HS-SPME



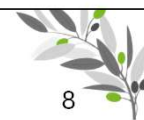
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Extraction Time

HTC, May 28th 2026

Damien Eggermont





Olive oil – Analytical techniques



- SPME different configurations/geometries

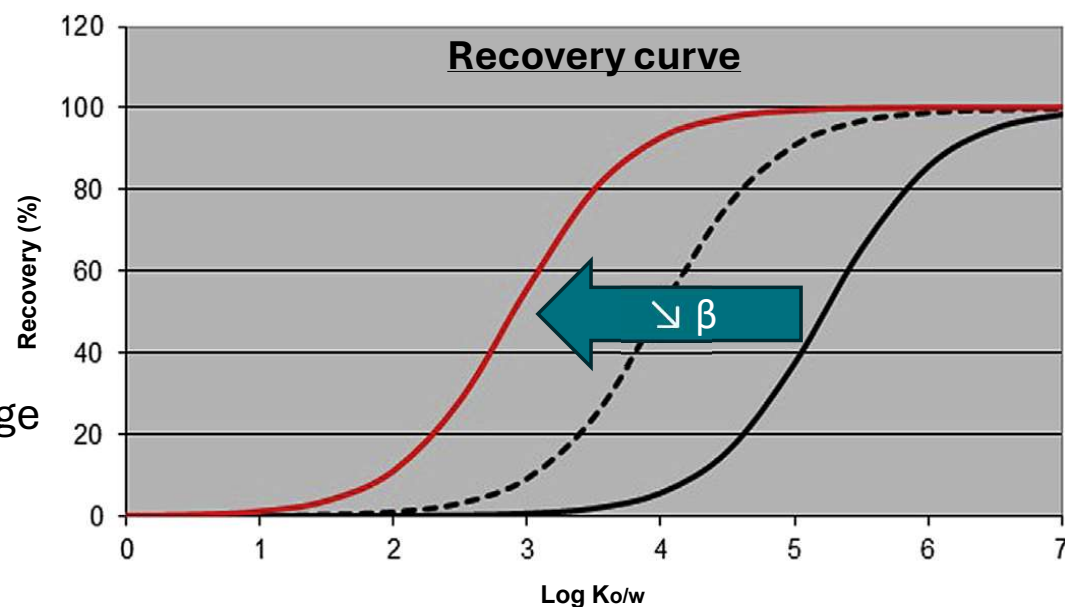
- HS-SPME
- Vac-HS-SPME
- HS-HiSorb
 - Higher extraction phase (S and V)
 - Higher recovery on broad polarity range

→ selectivity



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$$\beta = \frac{V_{sample}}{V_{extraction\ phase}}$$

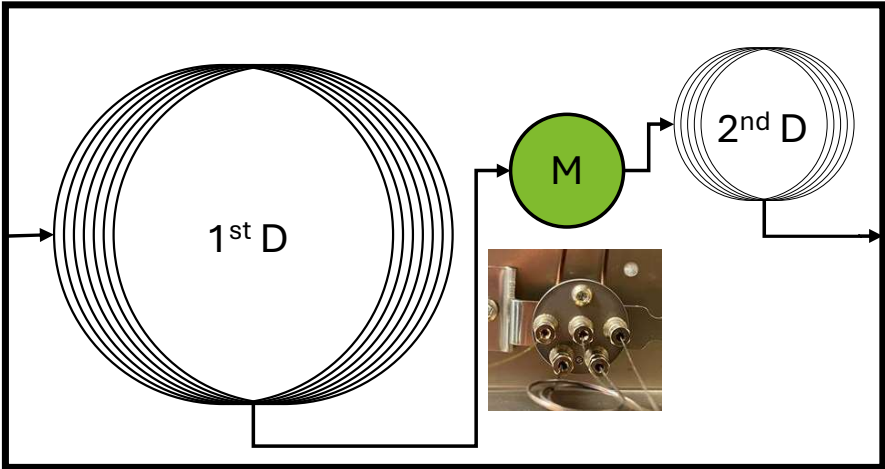
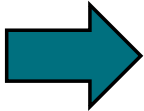




Analytical platform



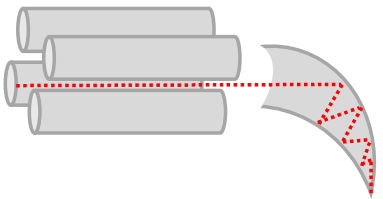
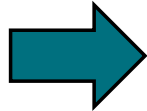
Sample Preparation



Apolar

Polar

GCxGC



qMS





Analytical techniques – Comparison



- SPME different configurations/geometries

• HS-SPME → conventional

✓ • Vac-HS-SPME → kinetics

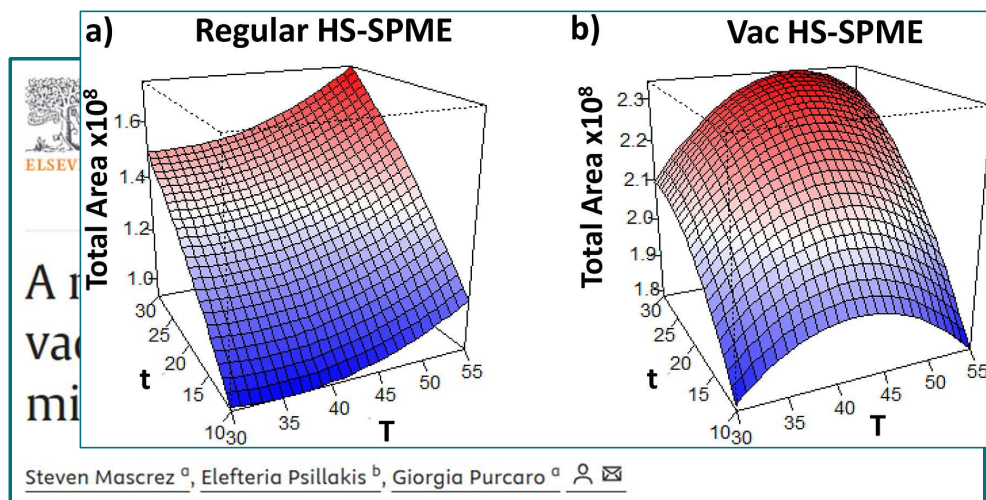
• HS-HiSorb → capacity

Comparison :

- Assessed VOO

- Small defects
- Positive attribute

• HS extraction optimisation



Conditions:

- 0.1 g of sample
- 45 °C
- 5 min preparation
- Extraction time : 20-30-45 min



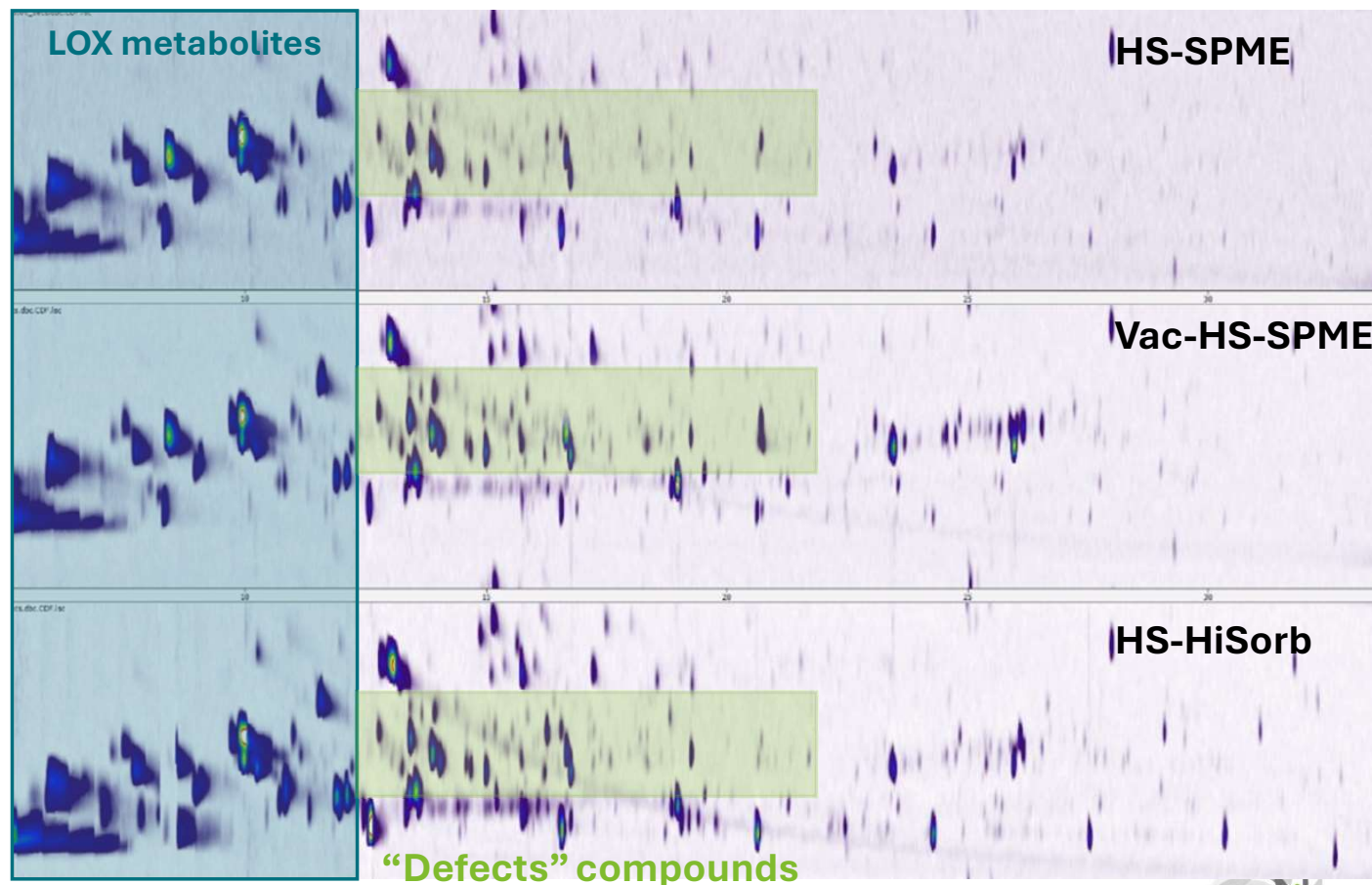
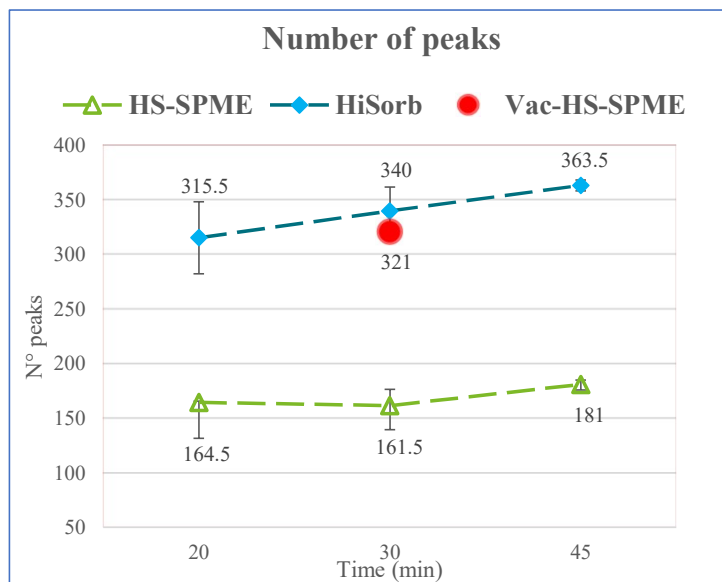


Analytical techniques – Optimisation



- SPME different configurations/geometries

- HS-SPME
- Vac-HS-SPME
- HS-HiSorb





Analytical techniques – Optimisation

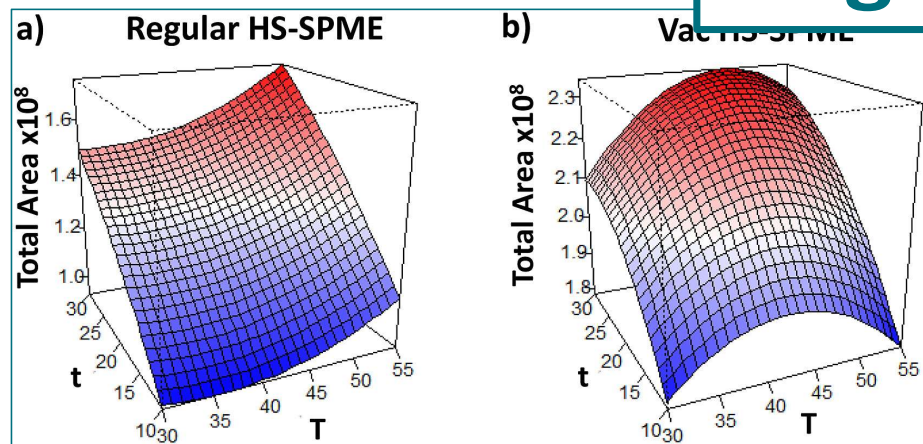


- SPME different configurations/geometries

- HS-SPME → 45 min

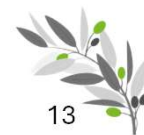
- Vac-HS-SPME

- HS-HiSorb → 45 min



Mascrez et al. (2020)

Higher = Better ?





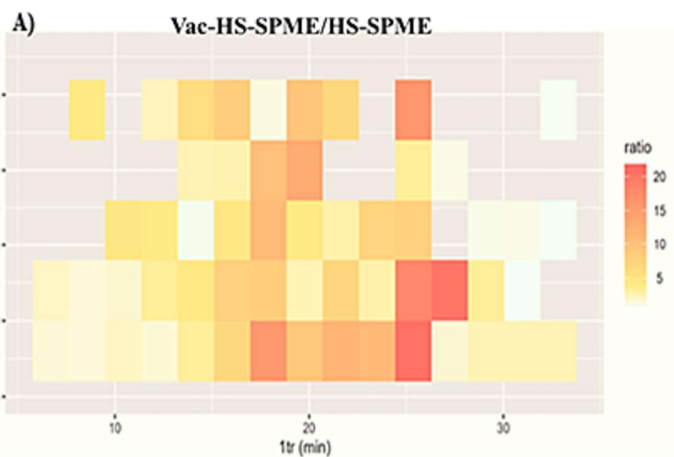
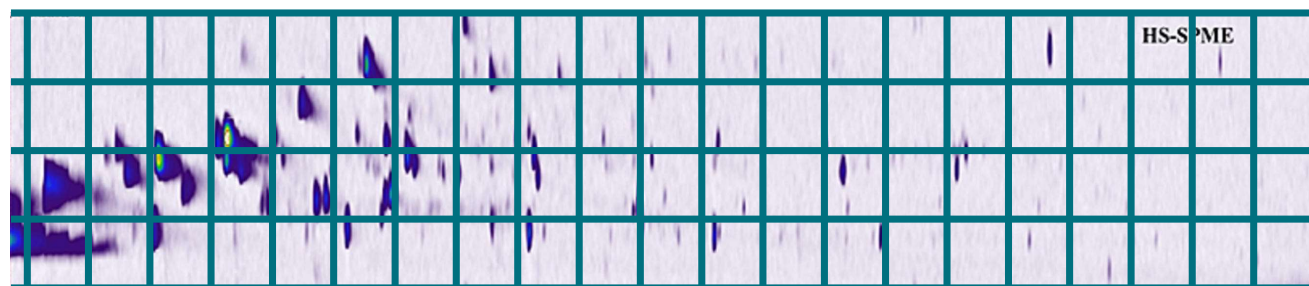
Analytical techniques – Comparison



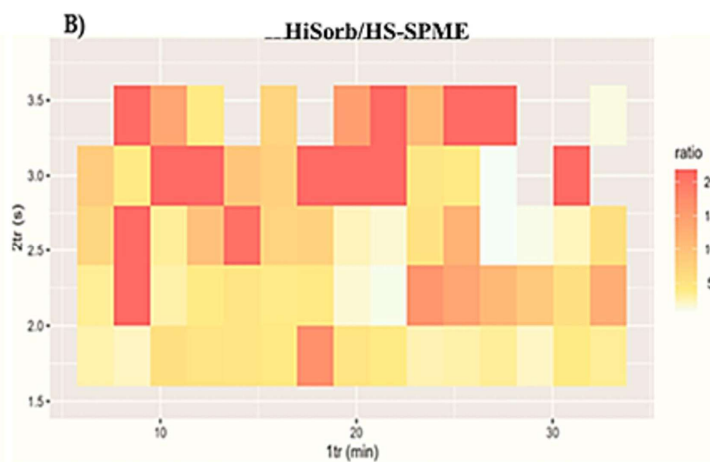
- SPME different configurations/geometries

- HS-SPME
- Vac-HS-SPME
- HS-HiSorb

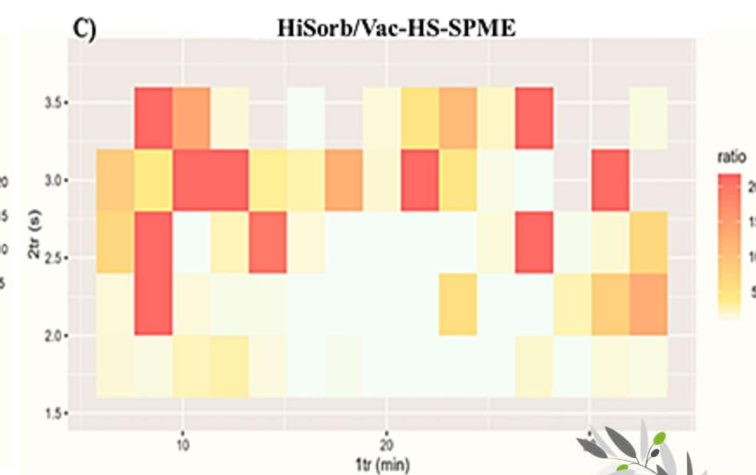
Mapping comparison



HTC, May 28th 2026



Digiglio et al. (2026)



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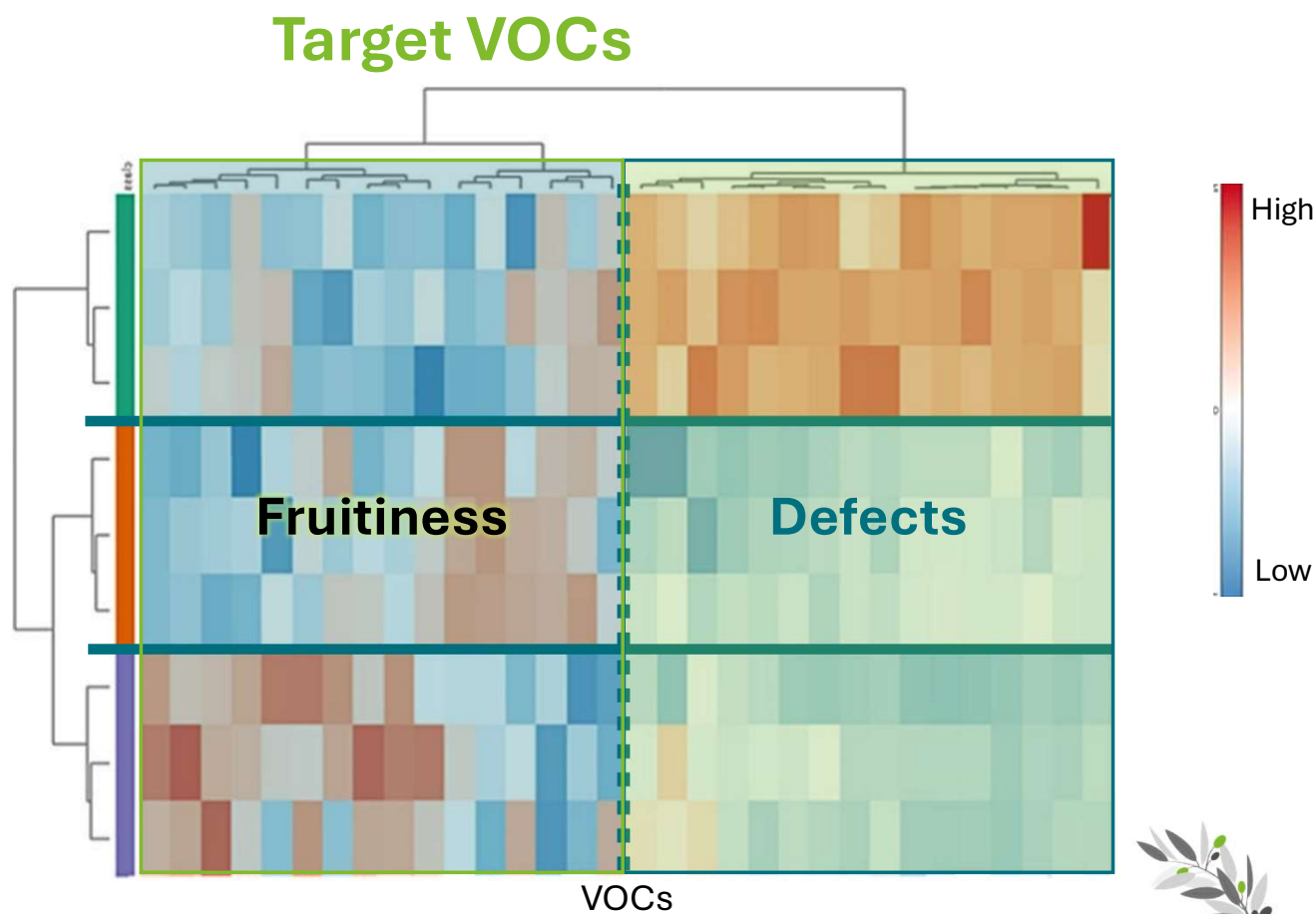


Analytical techniques – Comparison



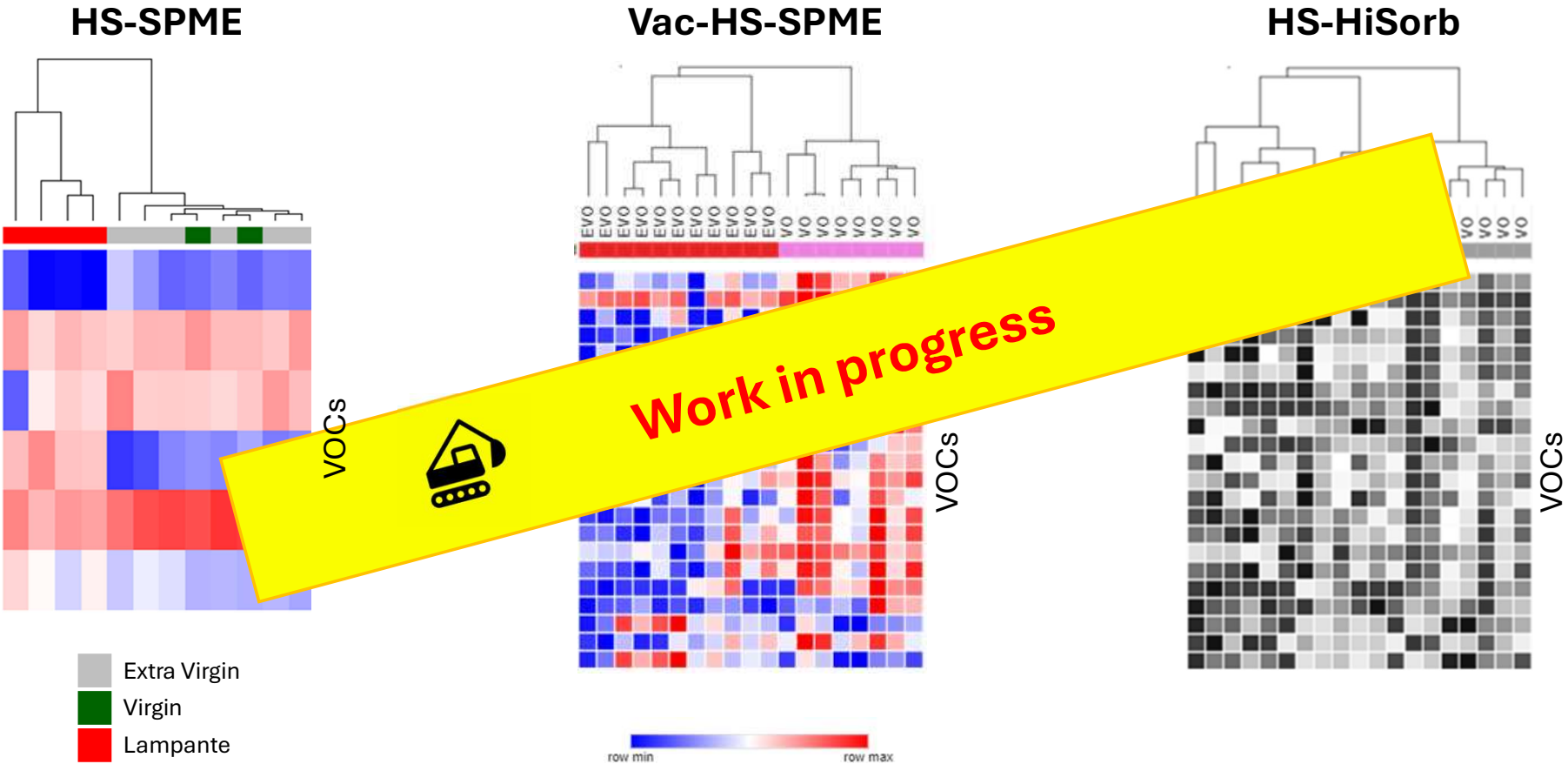
- SPME different configurations/geometries
 - HS-SPME 
 - Vac-HS-SPME 
 - HS-HiSorb 
- Clear distinction between techniques
 - Vac Vs. (HS-SPME & HS-HiSorb)
- Clear distinction between volatiles
 - Early eluting → Fruitness (LOX)
 - Late eluting → Defects

→ **Complementarity**





Classification performance





Conclusions and perspectives

- SPME different configurations/geometries

- Different selectivity

- (Highly) volatiles linked to positive attributes
 - Low volatiles linked to defect

→ HS-SPME or HS-HiSorb

→ Vac-HS-SPME

- Different global extraction yield

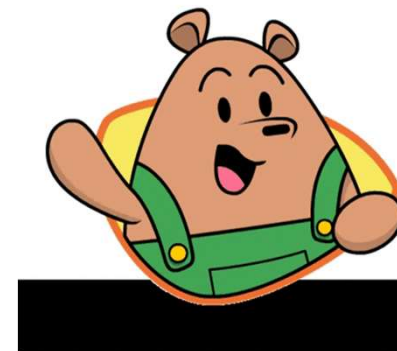
1. HS-HiSorb
2. Vac-HS-SPME
3. HS-SPME

Worth it ? Need it ?

- What about classification performances ?

- EVOO Vs. VOO ?
 - Geographical region ?
 -

SEE YOU LATER!





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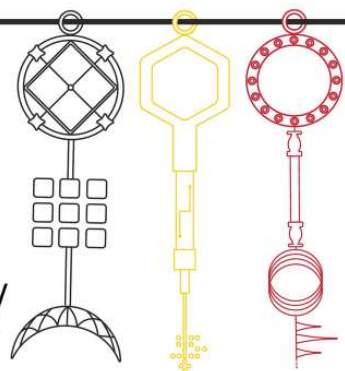


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