Application of GC-IMS to discriminate virgin olive oils according to their sensory grades

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OVERVIEW

Companies that sell virgin olive oils (VOOs) must often make quick decisions on the purchase and bottling of large lots of products for which the authenticity of the label-declared commercial category needs to be ensured. For this reason, the setting up of a rapid screening instrumental method to support the IOC Panel test able to discriminate the samples on the basis of their different quality grades (extra virgin olive oil EVOO, virgin olive oil VOO and lampante olive oil LOO) is an urgent need. In this preliminary work, 60 samples, belonging to different quality grades on the basis of sensory analysis, were analyzed by the GC-IMS. PLS-DA models were built using spectra regions and satisfactory results in terms of correctly classified samples were obtained.

INTRODUCTION

To date, the evaluation of the organoleptic defects in VOOs is carried out through sensory analysis, according to the method known as the IOC Panel test (COI/T.20/Doc. no. 3, 1987 and subsequent amendments).

The combination of results obtained by sensory analysis and instrumental methods is a matter of great concern; in fact, this approach can allow a rapid screening, supporting the sensory analysis by reducing the number of VOOs controlled for establishing their quality grade (Romero et al., 2015).

For these reasons, the qualitative and quantitative analysis of the profile in volatile compounds present in the headspace of VOOs, in particular by the SPME-GC-MS approach, has assumed great importance (Vichi et al., 2007).

In this work, a new interesting, sustainable and fast tool is represented by GC-IMS able to realize an aroma fingerprint for a possible discrimination of VOOs according to their quality grades (Garrido-Delgado et al, 2015).

METHODS

Sixty VOOs (12 EVOOs, 30 VOOs and 18 LOOs) were analyzed by using a gas chromatography coupled to an ion mobility spectrometer (GC-IMS) with a tritium source.

The samples, without any preparation step, were injected by a headspace device, after a thermoregulation at 40 °C for 20 minutes under agitation and examples of spectral data are shown in **Figure 1**.

Chemometric analysis

- Selection of the spectrum region giving greater information
- The spectra for each oil sample were arranged consecutively
- Obtained data set was 60 samples and 62645 variables (67 spectra in drift time for 935 variables in retention time)
- PCA was carried out to reduce the dimension of the dataset
- PLS-DA were developed to classify the samples according to sensory grades: EVOOs, VOOs and LOOs (Calibration and Full Cross Validation; for unequal class sizes the threshold was shifed)

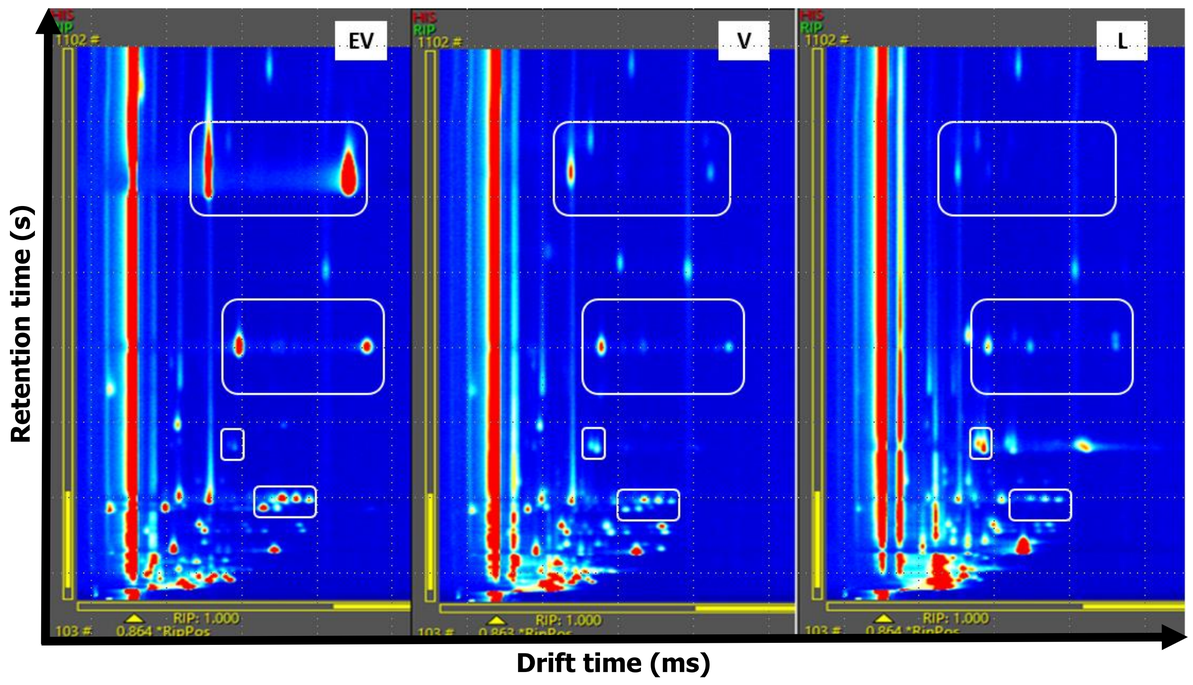

Figure 1 – Different spectral data of an Extra Virgin olive oil (EVOO), Virgin olive oil (VOO) and Lampante olive oil (LOO) with their main spots. In evidence the ones that decrease or increase in intensity throughout the three different categories.

RESULTS

PLS-DA models with 2 categories were sequentially elaborated:

- the first two models classified samples into **EVOOs** *vs* **NoEVOOs** (**Figure 2**) or **LOOs** *vs* **NoLOOs**
- the second two models classified NoEVOOs samples into **VOOs** *vs* **LOOs** and NoLOOs samples into **EVOOs** *vs* **VOOs**

PLS-DA results: samples correctly classified

	Calibration	Full Cross Validation
EVOOs	100%	100%
NoEVOOs	100%	75%
LOOs	89%	78%
NoLOOs	91%	81%
NoEVOOs		
VOOs	96%	90%
LOOs	83%	72%
NoLOOs		
EVOOs	100%	83%
VOOs	77%	73%

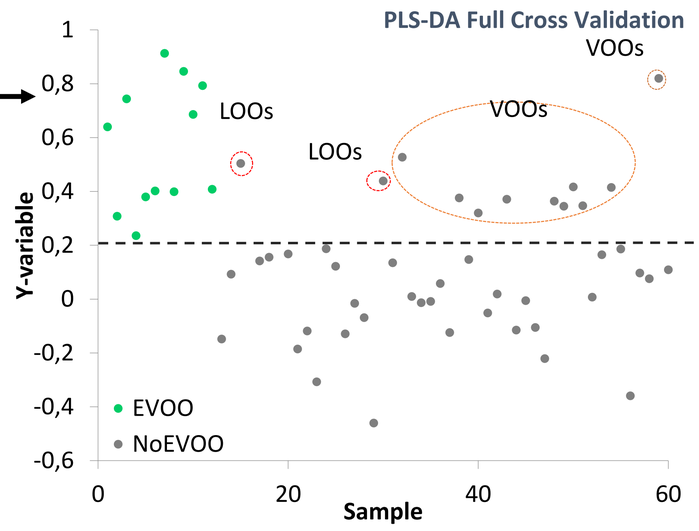

Figure 2 – PLS-DA Full Cross Validation for samples classified into EVOOs vs NoEVOOs.

CONCLUSIONS

This method evidenced promising results. In fact, it allowed to obtain reliable classification models (PLS-DA, sequential model) with a percentage of correct classified samples ranging from 77% to 100% and from 72% to 100%, for calibration and full cross validation, respectively. Satisfactory results were achieved discriminating EVOOs vs NoEVOOs (100% and 75%). Other samples will be analysed to improve the model robustness. Next steps will be to acquire information on the discriminating spots for each quality grade and secondly with which compounds they can be associated.

References

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