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^{13}C NMR Analysis for the Geographical Characterization of Extra Virgin Olive Oil (EVOO) Produced in Some Italian Regions

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Abstract

The geographical characterization of olive oils has been widely investigated by Nuclear Magnetic Resonance (NMR) spectroscopy. The matrices of data were built using integrals of the entire ^{13}C NMR spectrum as independent variables. The 49 signals deriving from the spectrum of each oil analyzed were compared through multivariate calibration methods in order to find common "patterns". The database of CREA-IT, corresponding to 1555 samples of EVOO (period 1995-1996) from several Italian regions (Abruzzo, Calabria, Lazio, Liguria, Puglia, Sardinia, Sicily and Tuscany), was processed. PCA (Principal Component Analysis) showed marked NMR lipid profile differences existing between EVOO samples from Calabria and Apulia, and between Liguria and Sicily, probably due to their different climate conditions. In fact, in geographically close Italian regions, with similar rainfall and temperature conditions (Lazio and Tuscany), it is not possible to discriminate EVOO samples because they show overlapping NMR lipid profiles.

Keywords: 13C NMR; Olive oil; Geographical characterization; Mono-varietal; PCA analysis

Introduction

Italy, located in the center of the Mediterranean Sea, is one of the most important olive producers. The extra virgin olive oil (EVOO) produced in some Italian regions is greatly appreciated worldwide for its attributes, which depend, among other factors, on geographical origin. The traceability and authenticity of olive oils have been widely investigated by Nuclear Magnetic Resonance (NMR) spectroscopy [1-3]. A new European Union regulation (EU 29/2012) has affirmed that "EVOO shall bear a designation of origin on the labelling" and that "the designation of origin shall appear on the packaging or on the label attached to the packaging". This directive is very important, because it recognizes that the agricultural practices and techniques used during the EVOO extraction procedure and the geographical origin are closely related to the qualitative characteristics of virgin olive oils. Knowing the origin of oil is therefore extremely important [4], and in recent years, various analytical techniques, in combination with multivariate statistical analysis (MVA) methods, have been applied for this purpose. Different techniques, such as IRMS and ^{1}H NMR, in combination with MVA, have been successfully used to discriminate Italian olive oils from Tunisian ones [5]. At the present time, the entire ^{1}H NMR metabolic profile of EVOO is considered strongly related to the geographic origin of the olive oil, as well as to its cultivar, maturation index, and/or technological factors [4,6]. In some studies, the combination of ^{1}H NMR fingerprinting and multivariate analysis has been successfully applied to predict the geographic origin of olive oils from different Mediterranean regions [7-9]. Resonance intensities of triacylglycerols obtained by ^{13}C NMR spectroscopy were used to classify olive oils from three production areas of the Puglia region [10].

EVOO database of the "Research Centre for Engineering and Agro-Food Processing" (CREA-IT)

In our work, the database of CREA-IT, corresponding to 1555 samples of EVOO (period 1995-1996) from several Italian regions (Abruzzo, Calabria, Lazio, Liguria, Puglia, Sardinia, Sicily and Tuscany), was processed. EVOO samples belonging to leccino, frantoio, piantone, sargano, moraiolo, mignola, raggiola, orbetana, ogliarola, carboncella and lavagnina cultivars are all characteristic of the examined regions. For each NMR sample preparation, 20mg of olive oil was exactly

weighed, dissolved in a volume of 0.9mL of deuterated chloroform (CDCl3), and transferred directly to a 5mm NMR tube. All the ^{13}C NMR spectra were recorded on a 499.84MHz spectrometer, operating at 11.7T (Varian NMR UNITY INOVA Narrow Bore, workstation UNIX-based Sun Microsystems, Varian NMR Instruments, Palo Alto, CA, USA). The matrices of data were built using integrals of the entire 13C NMR spectrum as independent variables. The 49 signals deriving from the spectrum of each oil analyzed were compared through multivariate calibration methods in order to find common "patterns". Statistical analysis was performed using specific MINITAB and IBM SPSS software. The selected variables were first subjected to analysis of variance (ANOVA) and then processed through the analysis of the main components (PCA). Thus, a data matrix was built, obtaining the plot of the scores, relative to each sample or loadings, relative to each variable. Each sample is positioned in the score plot with respect to the weight of the discriminating variables that characterize it. With this method we obtained a series of linear combinations that made it possible to maximize the separation between the samples. We therefore started to characterize the Italian productions in order to compare them with those of other countries.

Geographical characterization of mono-varietal EVOO from Calabria, Puglia and Sardinia

PC1 vs PC2 shows the distribution of samples in the score plot of the main components and discloses the differences/similarities between them (Figure 1). The different position of the samples coming from the regions of Calabria, Puglia and Sardinia, with respect to the weight of the discriminating variables, can be seen. In particular, along the first main component it is possible to distinguish the group of oils from Puglia compared to the Sardinian ones, while the second main component gives a good discrimination of the Calabrian oil group with respect to the other two groups.

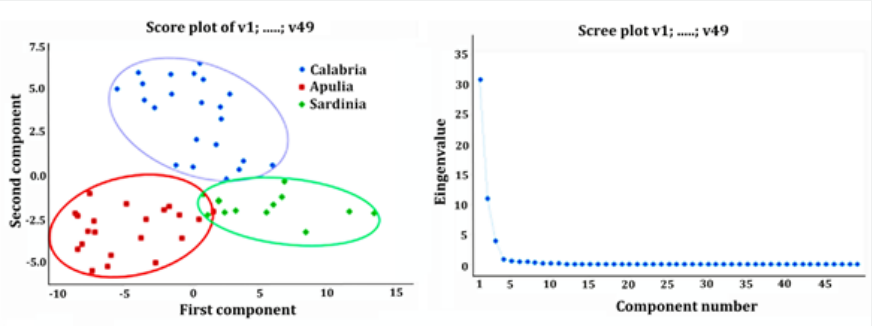

Figure 1: On the left the score plot for monovarietal EVOOs from Calabria, Apulia and Sardinia, and the related scree plot on the right.

Geographical characterization of mono-varietal EVOO from Abruzzo, Liguria and Sicily

PCA analysis for oils from other Italian regions, such as Abruzzo, Liguria and Sicily, discloses a significant discrimination of oil samples from the Sicily region, all of which are found to be positive along the first main component. On the other side, samples from Abruzzo and Liguria all had negative values of the first main component. The second main component discriminates the group of Ligurian oils compared to those coming from Abruzzo. In spite of a little overlapping of these two groups, differences between the samples of the three Italian regions can still be observed. In fact, PCA analysis shows that not only Italian regions such as Liguria and Sicily have distinct score plots, but also regions like Liguria and Abruzzo have oils that have a sufficient degree of differentiation (Figure 2).

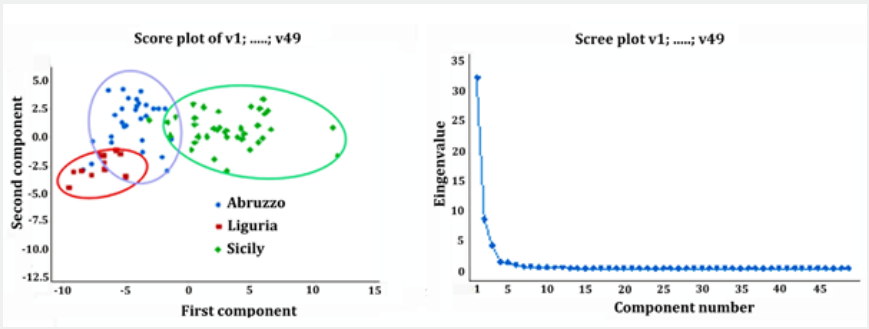

Figure 2: On the left the score plot for monovarietal EVOOs from Abruzzo, Liguria and Sicily, and the related scree plot on the right.

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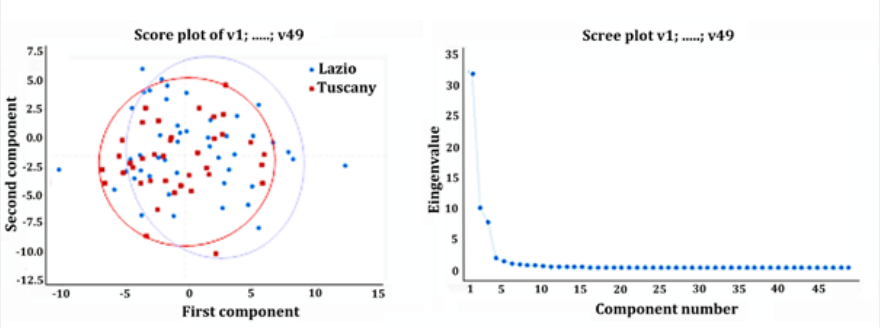

Figure 3: On the left the score plot for monovarietal EVOOs from Lazio and Tuscany, and the related scree plot on the right.

Geographical Characterization of Mono-varietal EVOO from Lazio and Tuscany

In this case, the PCA analysis of oil samples from the regions of Lazio and Tuscany shows an almost total overlap of the samples, which indicates that oils belonging to neighboring Italian regions have a certain degree of similarity (Figure 3).

Conclusion

This study shows that the similarities/differences between oil samples are influenced not only by variety of olive fruit but also by pedoclimatic characteristics of their native region. In fact, oils obtained from different varieties but from neighboring regions disclosed a significant similarity. The quality of EVOO and therefore its sensory characteristics, besides being dependent on the cultivar used for its production, is also influenced by climate (temperature, relative humidity of summer months, yearly rainfall), by crop practices and by oil extraction system [11]. In this work, ^{13}C NMR spectroscopy, combined with multivariate analysis techniques, allowed characterization of NMR lipid profiles of EVOOs, and their relationship with the native region. Marked differences existing between EVOO samples from Calabria and Apulia, and between Liguria and Sicily were observed. That could be due to their different climate conditions. In fact, in geographically close Italian regions, with similar rainfall and temperature conditions, it is not possible to discriminate EVOO samples because they show similar NMR metabolic profiles.

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