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GC×GC- TANDEM IONIZATION-TOFMS WITH COMBINED UNTARGETED AND TARGETED (UT) FINGERPRINTING OF VOLATILES FROM EXTRA VIRGIN OLIVE OIL: CHALLENGES FOR DATA ALIGNMENT AND RESPONSE NORMALIZATION

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Comprehensive two-dimensional gas chromatography (GC×GC) is the most effective multidimensional separation technique for in-depth characterization of complex fractions of volatiles (VOCs) in food. It enables highly informative fingerprinting and, if combined with Mass Spectrometry, it has the intrinsic potential to provide a detailed profiling giving access to the higher level of information encrypted in VOCs patterns: samples origin, technological signature, sensory profile [1,2].

Time of Flight Mass Spectrometry (TOFMS), featuring tandem hard and soft electron ionization, adds a further dimension to the analysis thereby enabling more confident identifications but also new challenges for the data processing. Consecutive alternate switching between hard (70 eV) and soft ionization (10-16 eV) provides complementary MS signatures (about each component and for each 2D pattern) increasing the dimensionality of the analysis and offering new possibilities for results cross-validation. However, suitable pattern recognition procedures (template matching and transformation algorithms) as well as correction/normalization of MS responses have to be considered to extend method fingerprinting capabilities over a wide-time frame.

This study focuses on the complex volatile fraction from Extra Virgin Olive (EVO) oils selected within the Italian Ager project “*Violin*” [3] because of their peculiar sensory characteristics and/or coherent with European Quality Labeling (Protected Denomination of Origin). VOCs are sampled by Head Space Solid Phase Microextraction (HS-SPME) with different sorption/adsorption polymers, separation is on a polar × semi-polar column combination and detection by tandem hard (70 eV) and soft (12 eV) electron ionization TOFMS. 2D patterns from EVO oils, acquired over a one-year time frame and under similar (but not identical) chromatographic conditions, are evaluated for 2D peak pattern shifts (targeted and untargeted peak-region features) and response fluctuations. Data transformation and normalization strategies are discussed in view of a fingerprinting method validation for large scale studies.

References

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