Multidimensional Gas Chromatography Hyphenated to Mass Spectrometry and Olfactometry for the Volatile Analysis of Citrus Hybrid Peel Extract

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Keywords: Multidimensional gas chromatography, Chirality, Sniffing port.

Investigating the composition of citrus hybrids is challenging because of the contribution of the traits of both parents. Research findings suggest that the peel oil of a somatic citrus hybrid is not the result of the addition of the parental traits (1). The resulting hybrids tend to produce the characteristic volatile compounds of both parents, but some compounds are over expressed, less expressed or even absent in the parents (2). GC-MS, which is the analytical technique of choice for the analysis of volatile constituents, combines specificity for target analysis, reliable identification for unknowns and good sensitivity. However, for many complex cases, the GC-MS system is not able to separate all the compounds of the chromatograms, and zones with coelutions can often be encountered. These zones can contain two or more compounds, which are difficult to identify and quantify. Different solutions have been developed for these analytical requirements, mainly as an attempt to overcome the critical aspects related to the separation of complex samples. Deconvolution procedure, based on sophisticated mathematical algorithms, are able to extract the mass spectrum of individual components in a mixture. Unfortunately, there are also limits to the contribution that deconvolution can provide. Consequently, heart-cutting multidimensional gas chromatography (MDGC) has for many years been recognized as a good tool for increasing chromatographic separation. The principle of this technique is to “heart-cut” the peak of interest from the first dimension (1D) into the secondary orthogonal dimension (2D) to obtain further separation. Recently, significant improvements have been made to access additional information by using the MDGC system. In the present work, Capillary Flow Technology (CFT) and Low Thermal Mass (LTM) GC columns, the most advanced technologies available in this area, were applied to perform heart-cutting MDGC analysis of complex regions of a citrus hybrid peel extract. The novel MDGC system provides many advantages over conventional systems, such as rapid heating and cooling and independent temperature control of the LTM-GC unit used as the second dimension (2D); low dead volume; high degree of inertness; and electronic pressure control of the Deans switch (DS), based on CFT. The main goal of this work was to investigate the volatile composition of citrus hybrids with not only a conventional approach based on GC-MS analysis using both non-polar and polar phases, but also with more advanced analytical techniques based on multidimensional GC (MDGC-MS/olfactometry [MDGC-MS/O] and enantioselective [Es]-MDGC-MS/O) for trace analysis.

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Acknowledgements: MEGA.

ISBN 978-85-66836-11-0