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Fast GC-MS/MS method for volatile flavour compounds analysis in oenological products: a promising tool to support the technological choices

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Summary: In this work a fast GC-MS/MS method to analyse the aromatic profile characteristic of the oenological products was developed. In a single chromatography run of 15 minutes, 50 volatile flavour compounds belonging of different chemical classes were identified and quantified.

Keywords: VFCs, GC-MS/MS, oenological products

Oenological products are one of the most complex chemical matrices, and among the other factors the aroma makes a crucial contribution to this complexity. Alcoholic beverages like wine and spirits contain a large number of flavour compounds belonging to several chemical classes with different polarities, volatilities and odour impact. The main chemical classes are higher alcohols, esters, aldehydes, lactones, terpenes, norisoprenoids, volatile phenols, fatty acids, carbonyls, sulphur and nitrogen compounds [1,2].

Some of these compounds are also present in the form of glycosidic conjugates (e.g. terpenes, norisoprenoids and phenols) that play an important role in aroma potential because of their possible conversion into the free counterparts through acidic or enzymatic hydrolysis during fermentation, fining and aging processes [3]. The rapid analytical measurement of free and glycosidic conjugates of volatile flavour compounds (VFCs) is essential to characterise the aroma of marketed wine and spirits, and moreover to support the technological choices during industrial transformation processes.

In this study, an innovative GC-MS/MS method useful for fast analysis of the broad aroma profile of oenological products was developed. Fifty volatile compounds of oenological interest belonging to the main chemical classes (acetates, acids, alcohols, aldehydes, esters, ketones, phenols, terpenes, and thiols) were identified and quantified in a single chromatographic run taking only 15 minutes.

Free and glycosidic conjugated VFCs were extracted by adsorption on a SPE cartridge (ENV+) and then eluted with dichloromethane and methanol respectively. Bound fraction was later hydrolysed with an enzymatic reaction and the free volatiles released were then extracted. Finally, both free and bound fraction were injected separately using the same GC-MS/MS method [4,5].

VFCs analyses was carried out using an Agilent Intuvo 9000 GC system coupled with an Agilent 7000 Series Triple Quadrupole MS equipped with an EI source (70 eV, 50 μ A). Separation was obtained injecting 2 μ l in split mode (1:5) into a 20 m DB-Wax UI (0.18 mm i.d. \times 0.18 μ m film thickness). The mass spectra were acquired in multiple reaction monitoring mode setting the instrument within the dynamic system [6].

The high sensitivity of the mass spectrometer, combined with accurate fast chromatography performed with a suitable capillary column, allowed us to avoid further concentration steps after solid phase extraction. Validation of the method was performed in relation to the limit of detection, limit of quantification, linearity range, repeatability, reproducibility, and recovery. Once validated, the method was successfully tested on commercial oenological samples (grape must, wine, balsamic vinegar and spirits), demonstrating its applicability to fast routine multiaroma analysis.

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