A fast gas chromatographic method for the study of semiochemical slow release formulations: validation by accuracy profiles and determination of UFM column performances

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Semiochemicals, which can be defined as chemical communication signals between living organisms, are more and more used in integrated pest management programs, acting as insect control or monitoring devices to reduce the pesticides treatments.

The application purpose of the present study consisted in verifying the protection efficiency of alginate gel beads towards incorporated semiochemicals. The procedure developed involved the quantification of sesquiterpene compounds in the formulations over time, when exposed to air and light, by means of a fast gas chromatograph coupled with a high frequency (300 Hz) flame ionisation detector.

E-*β*-farnesene, the alarm pheromone of many aphid species and *β*-caryophyllene, recently identified as the aggregation pheromone of the Asian ladybeetles *Harmonia axyridis* Pallas, are considered as attractants of aphid's predators and parasitoids.

The research focused first on the purification mode of the compounds by flash chromatography fractionation of essential oils with *n*-pentane as elution solvent. The solvent was evaporated with Büchi evaporator. *E*-*β*-farnesene was isolated at a purity of 83.8 % ± 0.3 % from the essential oil of *Matricaria chamomilla* L. The purity of *β*-caryophyllene extracted from *Nepeta cataria* L. essential oil was of 97.7 % ± 0.5 %.

Secondly, the fast chromatographic method optimised, in terms of resolution, for the sesquiterpenes analysis was completely validated by means of the accuracy profile concept based on the guidelines of the Société Française des Sciences et Techniques Pharmaceutiques (SFSTP). The accuracy profiles were constructed for the two analytes (*E*-*β*-farnesene and *β*-caryophyllene) by considering a risk of 5 % and a linear regression model. The different validation criteria were evaluated and the lowest limits of quantification were determined.

The sample capacity and the UFM (Ultra Fast Module) column efficiency were also evaluated respectively, with the evolution of the number of theoretical plates in function of the amount of sample injected, and with the Van Deemter plots.