**COUPLING OF VAC-HS-SPME AND GC×GC-QMS FOR SIMULTANEOUS 5-HMF QUANTIFICATION AND VOLATILE PROFILING IN HONEY**

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**Max 250 words!**

Faced with the growing complexity of the food industry's demands, analytical techniques are evolving towards increasingly sophisticated and multi-response analyses. In this respect, multidimensional techniques such as GC-MS and GC×GC-MS are ideally suited.

Regarding sample preparation, headspace-solid-phase microextraction (HS-SPME) is one of the most favoured techniques for food volatile analysis thanks to its simplicity and ability to concentrate a wide range of compounds without needing solvents. However, the extraction of low-volatile compounds can be limited. Among different available strategies to increase their extraction, vacuum-assisted (Vac)-HS-SPME is highly promising. The use of vacuum facilitates the volatilisation of less-volatile compounds by lowering the gas-phase resistance to the mass transfer while maintaining the same extraction efficiency for the more volatile compounds.

In this project, Vac-HS-SPME has been coupled with GC×GC-qMS to quantify the 5-HMF (storage and heat processing marker) and analyse the volatile profile of honey. 5-HMF is regulated by the EU (2001/110/EC) fixing the maximum limit level to 40 mg/kg in most cases. Validation of the proposed method has been realized using a matrix-matched calibration, reaching LOD and LOQ of 1.6 and 4.7 mg/kg, respectively; while a recovery of 98% and a RDS of 21% were achieved. The method was trailed with eight real-world samples against the official HPLC method showing, an average bias of 6%. In terms of greenness the proposed method gave better results using the AGREE metrics, while the practicality was similar, as calculated with the BAGI.