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APCI and HESI source evaluation to investigate nitrosamine formation in meat

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Summary: *Meat is a very complex matrix and the legal limits for some nitrosamines are extremely low (2 ug/kg). A method with adequate cleaning of the matrix and maximum sensitivity in ionization was then evaluated using an APCI – HESI source couple with a HQOMS.*

Keywords: *meat, nitrosamine, APCI, HESI, Orbitrap*

Introduction

Nitrosamines are known as carcinogens and could potentially formed in cured meat as result of the reaction of amines with nitrites, which are generally used as a preservative [1].

Experimental

In this study 4 samples of hums have been produced differently in order to encourage or not the development of nitrosamines. Various extractions were tested to find the best recovered of these compounds and the best matrix purification using acetonitrile:acetone (50:50, *v/v*) and EXtrelut NT combined with Florisil. Different columns were tested for correctly separate the compounds, finding in the use of Acclaim Vanquish 2.7 µm PA2 2,1x150 mm the best choice. As eluents water and acetonitrile with the 0.1% of formic acid were used in gradient and a sample SPE on-line pre-treatment was performed to also allow the injection of 100 µL. APCI source and HESI source, in positive and negative mode, were tested to evaluate the most performing ionization and high resolution mass spectra were acquired, scanning from *m/z* 50 to 500, in profile mode through full MS-data dependent MS/MS analysis (resolution 70,000 FWHM for m/z 200, 3 Hz). The maximum injection time (IT) was set at 100 ms and the automatic gain control (AGC) target at $3 \cdot 10^6$ ions. Data-dependent mass spectra were collected at a resolution of 17,500 FWHM (defined for m/z 200, 12 Hz, IT of 50 ms, AGC target of $1 \cdot 10^5$ ions).

Results

N-Nitrosodimethylamine (DMNA), N-Nitroso-N-methylethylamine (NMEA), N-Nitrosopyrrolidine (NPYR), N-nitrosodiethylammine (NDEA), N-Nitrosopiperidine (NPIP), N-Nitrosomorpholine (NMOR), N-Nitrososarcosine (NSAR), N-Nitrosodi-N-propylamine (NDPA), N-Nitroso-L-Proline (NPRO), N-Nitrosodi-N-butylamine (NBUT), 3-Nitroso-4-thiazolidinecarboxylic Acid (NTCA), N-Nitroso-2-methylthiazolidine-4-carboxylic Acid (NMCA), N-Nitrosonornicotine (NNN), N-Nitrosodiphenylamine (NDPhA), 4-(N-Nitrosomethylamino-1(3-pyridyl)-1-butanone (NNK) were tested. In solvent only NSAR best respond with APCI interface with a LOD of 0.2 µg/L while in HESI with a LOD of 2.5 µg/L. In meat

matrix analysis instead, the nitrosamines DMNA, NSAR, NMOR, NPRO, NMEA, NTCA, NDEA, NMCA and NPPI responded with 0.5-2 orders of magnitude higher, NPYR, NDPA and NBUT had no variation and only NNN, NNK and NDPhA showed a minor response.
In only one ham sample was found nitrite concentration up the LOQ, but nitrate was always present. Finally, the sample with nitrite was the only one the showed nitrosamine concentrations higher than LOD, NSAR at 6 µg/kg, NTCA at 140 µg/kg and NMCA at 403 µg/kg.

Conclusions

The HPLC-HQOMS has proved to be an excellent technique for the analysis of non-volatile and volatile nitrosamines. To evaluate all the tested nitrosamines with a low LOD both the source APCI and HESI must be use.

References

1. Lehotay, S. J., Sapoahnikova, Y., Han, L., Johston, J. J., Journal of Agriculture and Food Chemistry (2015), 63 (47), 10341-10351.