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Title:

Prediction of coffee sensory quality in cup by Headspace Solid Phase Microextraction-Mass-spectrometry-based electronic nose (HS-SPME-MS).

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Prediction of coffee sensory quality in cup by Headspace Solid Phase Microextraction-Mass-spectrometry-based electronic nose (HS-SPME-MS).

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Summary: *Headspace Solid Phase Microextraction (HS-SPME) directly coupled with mass spectrometry (MS) based on electronic nose is a promising technique as an analytical decision maker tool in routinely controls to define the coffee sensory quality in cup.*

Keywords: *Coffee sensory quality prediction, analytical decision maker, HS-SPME-MS*

1 Introduction

Aroma is a primary hedonic aspect of a good coffee playing a fundamental role in the coffee choice [1] and can be considered as a signature of the products [2-6]. The cup tasting is nowadays the most important criteria to define the coffee quality, nevertheless it is time-consuming in terms of panel training and alignment. Defining a relationship between the chemical profile and the aroma sensory impact is an important challenge both in analytical and in industrial fields because it can afford food industries to obtain an objective evaluation of their products. The aim of this work is to use diagnostics mass spectral fingerprints in developing an instrumental prediction model which can be exploited as an analytical decision maker in routine controls to define coffee sensory quality in cup.

Coffee samples were sensorially evaluated through monadic profiling and analyzed by HS-SPME-MS and data elaboration was carried out with multivariate analysis.

HS-SPME is a reliable high-concentration-capacity technique easy to automate, that can directly be combined online with mass spectrometry. Non-separative MS methods, better known as mass spectrometry-based electronic nose or MS-nose, provide a representative, diagnostic, and generalized mass spectrometric fingerprint of the volatile fraction of a sample, analyzed directly without prior chromatographic separation, in which each m/z ratio acts as a "sensor" whose intensity derives from the contribution of each compound producing that fragment. These methods, in combination with sensometrics, can be completely implemented in an automatic TAS technology (Total Analysis System) giving a high throughput solution for quality control.

2. Experimental

Samples: One hundred and Twenty coffee samples with distinctive sensory notes, originating from different countries were analysed. Roasting degree was set at 55°Nh, to be close to the international standardization protocol for cupping (SCAA, 2015).

Descriptive sensory analysis of coffee aroma

The samples were submitted to sensory evaluation through quantitative descriptive analysis by a coffee panel experts following the SCAA protocol. Cup quality was assessed for several attributes: flowery, fruity, woody, nutty, spicy, acidity, bitterness. Quality and intensity of each attribute were evaluated simultaneously, upon a scale from 0 to 10.

Head Space Solid Phase Micro Extraction sampling: Volatiles were sampled by HS-SPME using an MPS-2 multipurpose sampler (Gerstel, Mülheim a/d Ruhr, Germany) online integrated with an Agilent 7890 GC coupled to a 5975 MS detector (Agilent, Little Falls, DE, USA). 1,5 g of ground roasted coffee in a 20 mL vial were sampled by HS-SPME at 50 °C for 10 min. SPME fiber: PDMS/DVB d_f 65 µm, 1 cm long (Supelco, Bellefonte, PA, USA).

MS-Nose instrument set-up: oven and injector temperature, 250 °C; injection mode, split; split ratio, 1/10; carrier gas, helium; flow rate, 0.4 mL/min; fiber desorption time and reconditioning, 3 min; transfer column, deactivated fused silica tubing (d_c = 0.10 mm, length = 6.70 m). MSD Conditions: ionization, EI mode at 70 eV; transfer line, 280°C.

Standard tuning was used, and the scan range was set at m/z 35−350 with a scanning rate of 1.000 amu/s.

Data acquisition and elaboration Data were acquired and processed with an Agilent MSD Chem Station ver. E.02.01.1177. Chemometric analysis were performed with Pirouette software ver. 4.0 (Infometrix, Inc., Bothell, WA, USA).

3. Results

Instrumental data treatment The HS-SPME-MS patterns provide a fast but diagnostic response. The significance of the TIC is hidden because the intensity of each fragment (m/z) is the sum of more than one chemical compound at different concentrations (Fig.1). In this case, the use multivariate analysis to "extract" significant information from the MS profile is mandatory. The original MS spectral fingerprints were normalized versus the most intense ions taken as 1; each m/z intensity value is expressed as a percentage of the intensity of the base m/z fragment and baseline corrected.

Partial Least Square Discriminant analysis (PLS-DA) was used to select variables (m/z) able to describe high and low scores samples within a sensory attribute, reducing the noise. 36 ions resulting as the most discriminating out of 315 for all the sensory notes investigating.

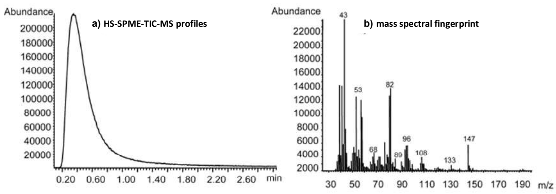
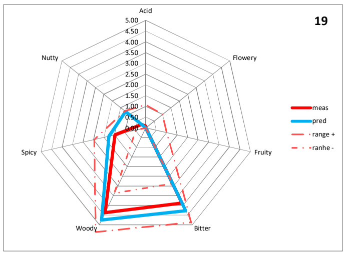


Figure1HS-SPME-MS profile A) and b) the resulting mass spectral fingerprint

Sensory data treatment All the samples were submitted to a sensory evaluation by five experts. A 1-way ANOVA with all the scores of the five experts and a paired t-test between each expert was performed. Experts who had high variance in relation to others were removed and averages were used as ‘‘main scores’’ for the seven attributes under study. **Regression model** To build the regression models a training set (146 objects) and an external test set (30) were used. Leave-p-out cross-validation (20) was the method used to select the number of components in the models Partial Least Square regression (PLS).

In spite of the high variability of the samples acid, bitter and woody notes were the most reliable.

Figure2 Samples aroma profiles comparison between sensory evaluation and sensory prediction



The mean error in the sensory scores prediction on test set with these data is within the fixed limit of ±1. While for other notes e.g. spicy, fruity and flowery notes show a good fitting between chemical and sensory evaluation. However, for these last the models present a low predictive ability because of both a) the high noise due to an unbalanced pool of samples within the scores range, and b) the difficulties linked to a too general lexicon used to define the notes.

4. Conclusions

The results show that the HS-SPME-MS fingerprints in combination with sensometrics is a promising technique to be used as a TAS system for an high throughput solution to define the coffee sensory quality in cup. To be reliable in sensory scores prediction, it requires a robust mathematical model derived from a high number of representative samples and an accurate alignment in the lexicon to rate the samples.

References

1.Sunarharum, W. B., Williams, D. J. & Smyth, H. E. *Food Res. Int.* **62,** 315–325 (2014).

2.Ruosi, M.R., Liberto, E., Cordero, C., Cagliero, C., Rubiolo, P., Bicchi, C., Sgorbini, B. *J. Agric. Food Chem.* **60**, 11283-11291 (2012).

3.Liberto, E., Ruosi, M.R., Cordero, C., Rubiolo, P., Bicchi, C., Sgorbini, B. *J. Agric. Food Chem.* **61**, 1652-1660 (2013).

4. Folmer, B. *Food Res. Int.* **63,** 477–482 (2014).

5.Ribeiro, J. S., Augusto, F., Salva, T. J. G., Thomaziello, R. A. & Ferreira, M. M. C. *Anal. Chim. Acta* **634,** 172–179 (2009).

6.Lindinger, C., Labbe, D., Pollien, P., Rytz, A., Juillerat, M. A., Yeretzian, C., Blank, I. *Anal. Chem.* **80**, 1574–1581 (2008).