MULTIVARIATE ANALYSIS BASED ON HS-SPME/GC-MS FINGERPRINT AND VOLATILE COMPOSITION FOR THE CHARACTERIZATION OF EXHALED HUMAN BREATH

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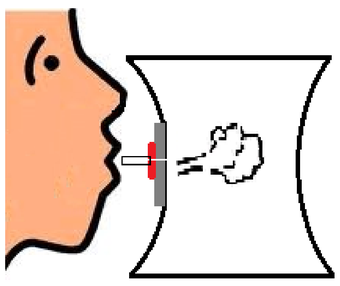
INTRODUCTION

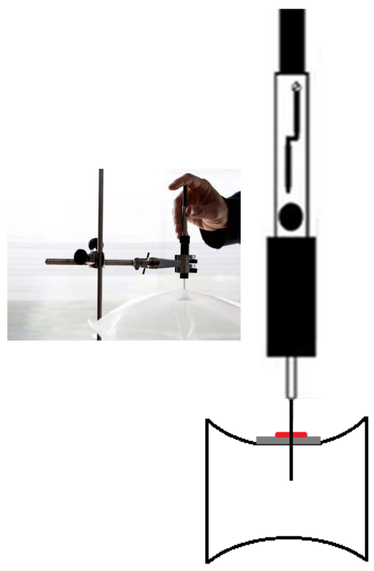
This study aims to exhaustively identify the volatile organic compounds (VOCs) that can appear in exhaled human breath and determine their relative composition using Gas Chromatography/Mass Spectrometry (GC-MS) with Solid-Phase Microextraction (SPME)[1], in order to choose the appropriate coating to functionalize an array of sensors for electronic nose[2]. The first step was to compare human breaths in order to detect target VOCs that can differentiate human breaths.

Breath capture and analysis method

16 healthy subjects (smoker and non-smokers of all ages and gender) who had **not ingested coffee or alcohol for at least 24h** inhale moderately and then to exhale as much as possible.

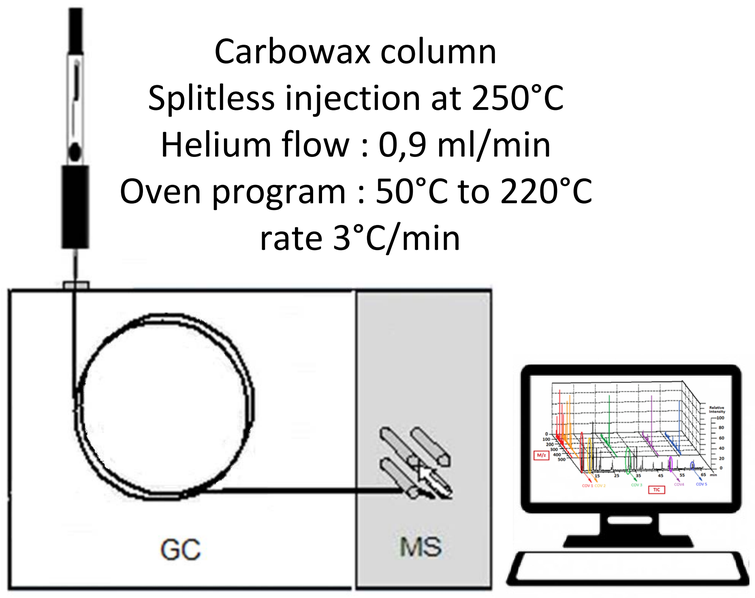


Tedlar bags (5L Tedlar® PLV Gas Sampling Bag w/Thermogreen® LB-2 Septa) were used to collect breaths



SPME fibers (Carboxen/Polydimethylsiloxane) were used to extract and concentrate human breath VOCs. In the same time, a second fiber was used to analyze ambient air (as blank)

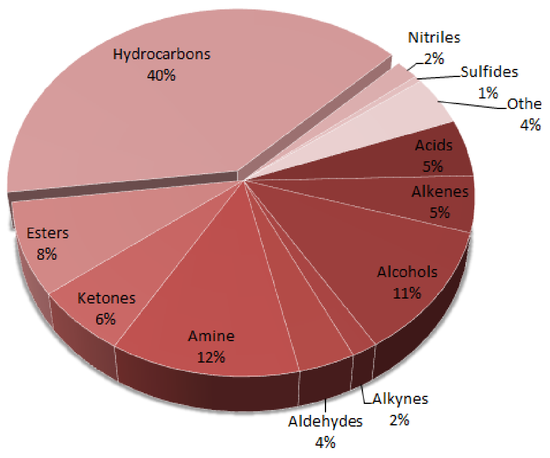
Carbowax column
Splitless injection at 250°C
Helium flow : 0,9 ml/min
Oven program : 50°C to 220°C
rate 3°C/min



GC-MS (Agilent with quadrupole mass analyzer was used to identify breath VOCs

Results - VOCs identification

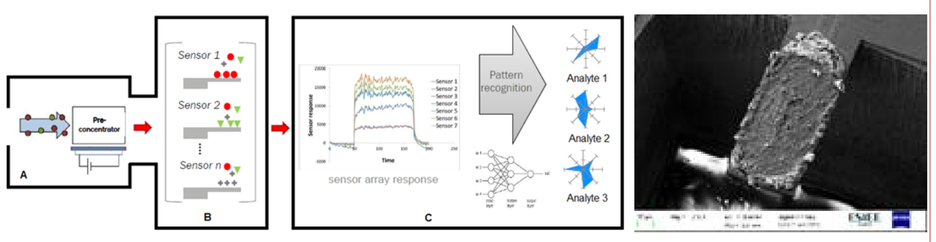
2500 VOCs were detected on breath samples like hydrocarbons alcohols, aldehydes, ketones, amines, esters...



Volatiles compounds family repartition

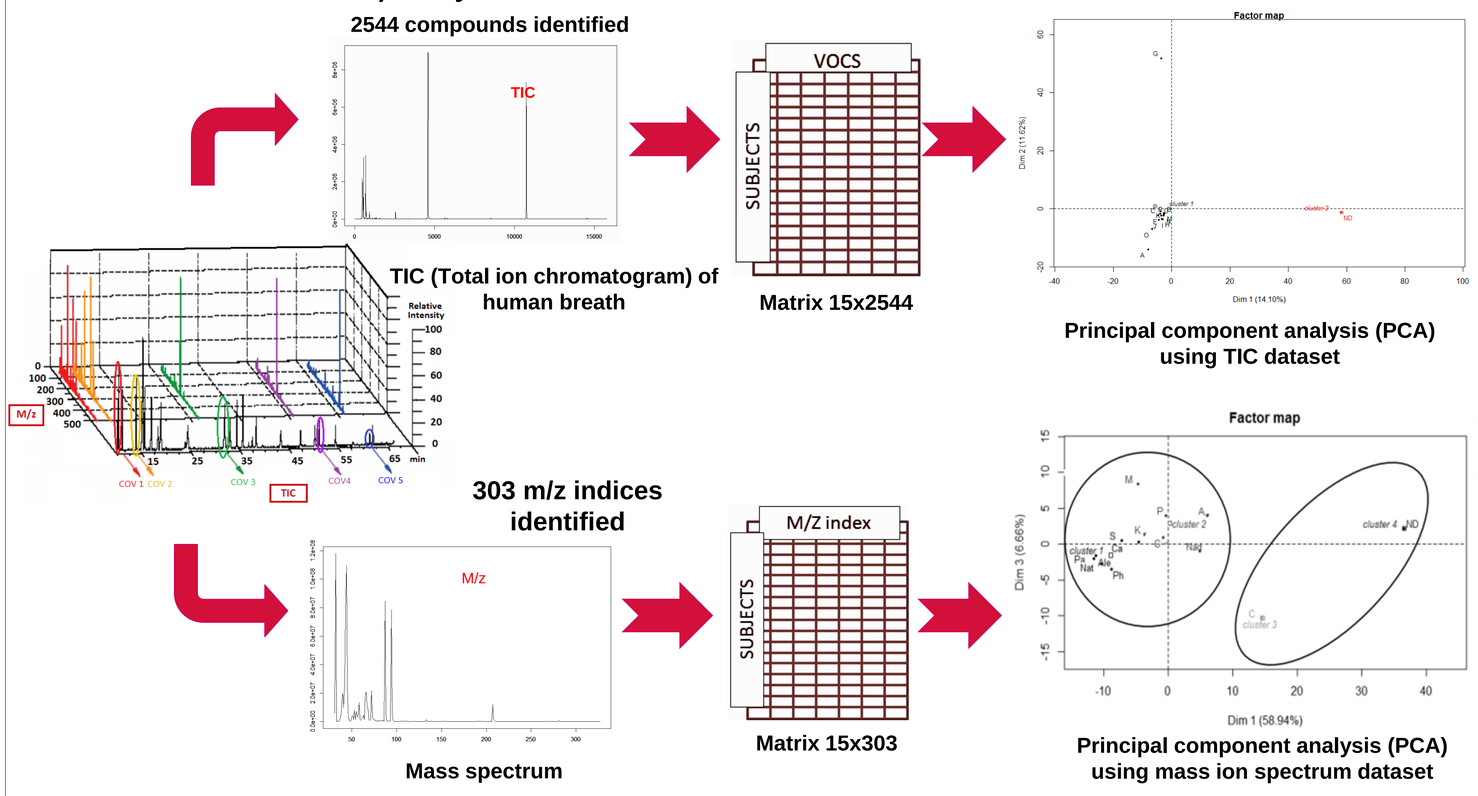
Results - Perspectives for e-Nose applications

An electronic nose is a device that attempts to mimic the mammalian senses using sensor arrays and pattern recognition systems. The HS-SPME/GC-MS fingerprint analysis made possible the identification of VOCs on human breath. Results of this analysis are crucial on the choice of best sensitive layer for sensors functionalization.



Results - Multivariate statistical treatment

Multivariable PCA over total ion chromatogram (TIC) and over mass ion spectrum have been done in order to classify subjects. Both methods are illustrated bellow:



In the first method, each sample is characterized for the number and type of volatile organic compounds found by the GC-MS analysis. The Total Ion chromatogram (TIC) is used to compose the matrix subjects x VOCs which is used for proceed to the principal component analysis (PCA). Results of this analysis have not been of help in the discrimination of relevant groups. In the second method, each sample is characterized regarding the number of scores m/z found by the GC-MS analysis. To extract this information, we used the package "XCMS" of R®. The package implements first, a data filtering, detection of pics, then a deconvolution and finaly, the alignement of detected pics. Each m/z value can be associated, for example, to a carbon length. The resultant matrix, smaller and pre-treated, is analised using a PCA. Results of this analysis leads to a separation of subjects into two groups : smokers and non-smokers.

CONCLUSIONS

The capture and analysis method **(HS-SPME/GC-MS)** is robust and allows to detect more than 2500 VOCs in human breaths. The XCMS treatment prior to the application of a statistical method like PCA seems to improve the quality of the discrimination of subjects and allows to distinguish two groups : smoker and non-smoker subjects. Results of human breath characterization will be considered in the choice of sensitive layers for e-nose sensors and XCMS+PCA method will be integrated as part of pattern recognition algorithm of the e-nose system.

References: [1] Ouyang, G., & Pawliszyn, J. (2006). SPME in environmental analysis. Analytical and bioanalytical chemistry, 386(4), 1059-1073.
[2] Possas, M., Rousseau, L., Ghassemi, F., Lissorgues, G., Scorsone, E., Manai, R., & Bergonzo, P. (2015, April). Frequency profile measurement system for microcantilever-array based gas sensor. In Design, Test, Integration and Packaging of MEMS/MOEMS (DTIP), 2015 Symposium on (pp. 1-5). IEEE.

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