Analysis of aldehydes released by rice flour using sorbent adsorption and thermal desorption - gas chromatography/mass spectrometry : *Development and validation*

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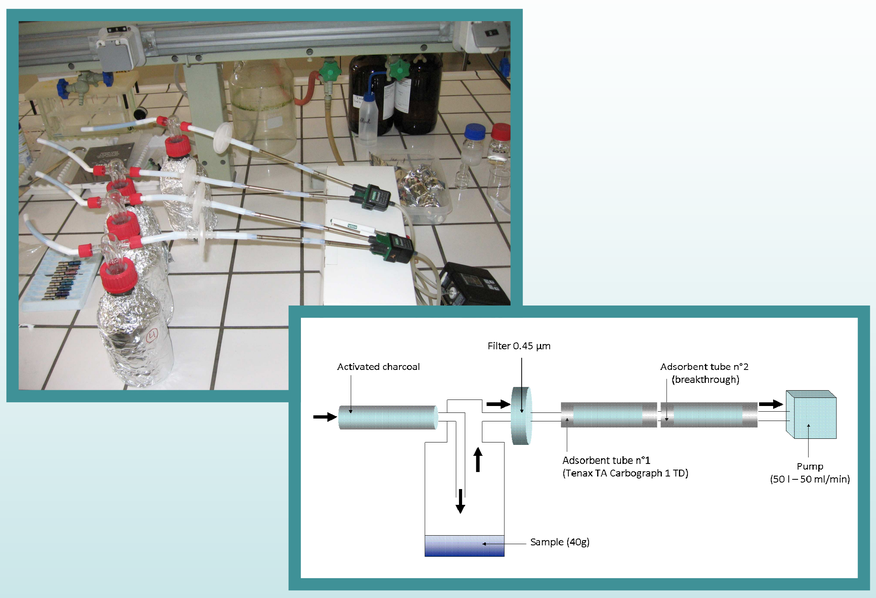
Introduction

Odor is an essential characteristic and a quality marker in baby foodstuff, like a partially hydrolyzed and pregelatinized rice flour. The rice flour studied by active sampling and TD-GC/MS highlighted the presence of three aldehydes: Hexanal, Octanal, Nonanal. These compounds are known to be caused by lipid oxidation, one of the major alteration reactions in food. The method was developed and validated on a large range for the three aldehydes. It was then applied to the rice flour to determine if it can be used to follow the effect of ageing on the aldehydes production.

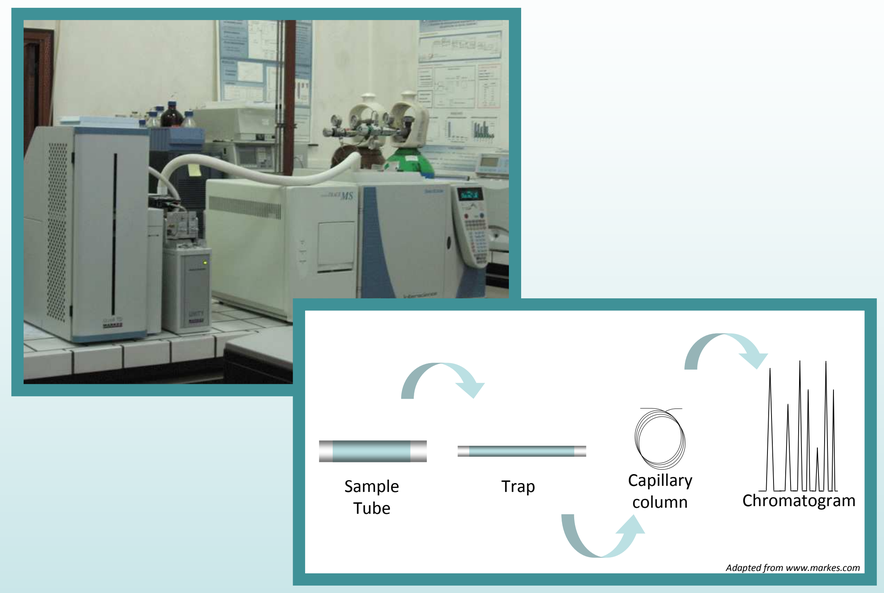
Experimental

Material and Method

Sampling



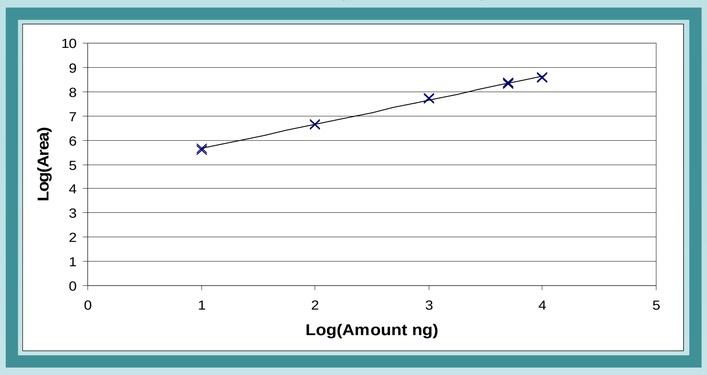
TD-GC/MS



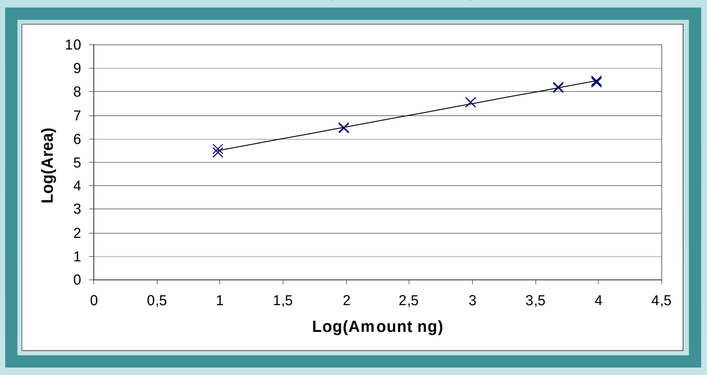
Method Validation

Calibration curves

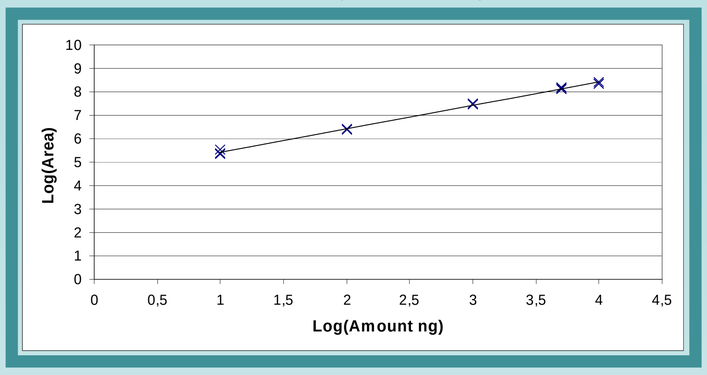
Hexanal *(R^2 = 0.9978)*



Octanal *(R^2 = 0.9973)*



Nonanal *(R^2 = 0.9964)*



Validation parameters

Compounds	Hexanal	Octanal	Nonanal
Retention time (min)	14.17	22.37	26.02
Range (ng)	0-10000	0-10000	0-10000
R^2	0.9978	0.9973	0.9964
LOD[a] (ng)	0.3	11.9	3.5
LOQ[b] (ng)	0.5	23.8	7.0
Accuracy[c] (%)	0.25-7.44	0.17-2.56	0.85-4.26
RSD[d] (%)	0.90	0.77	1.22
Recovery[e] (%)	99.93-100.00	99.39-99.65	99.68-99.84

[a] Limit of detection (LOD) was expressed as three times the standard deviation of the blank value

[b] Limit of quantification (LOQ) was expressed as six times the standard deviation of the blank value

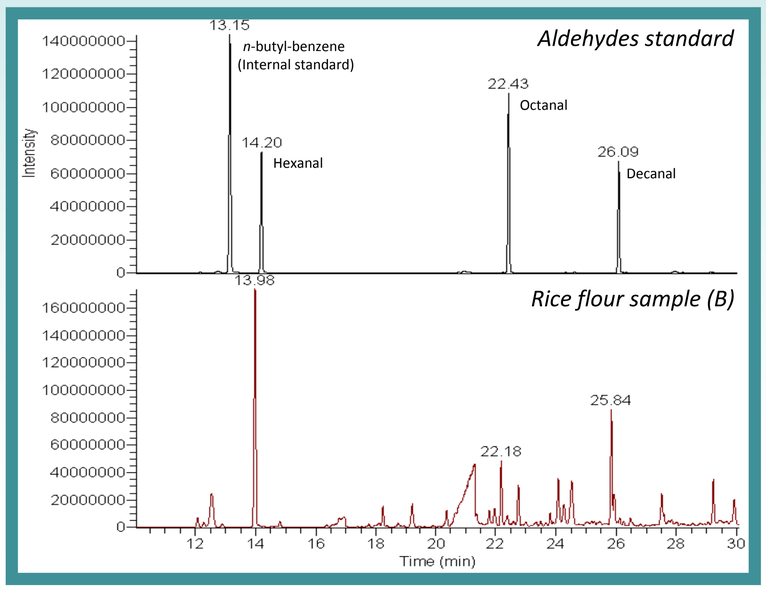
[c] Accuracy was measured for five levels (10 – 100 – 1000 – 5000 – 10000 ng)

[d] Repetability was measured for five repetitions of 1000 ng

[e] Recovery was defined as the % area for the first desorption compared with the two successive desorptions, for three tubes with three levels (1000 – 5000 – 10000 ng)

Rice flour samples

TD-GC/MS chromatograms of an aldehydes standard mix to 1000 ng on a tube and a rice flour sample



Samples description

Compounds	Sample A	Sample B		Sample C	
Repetitions	*n = 1*	*n = 2*		*n = 2*	
Hexanal	37.8	176.9	181.8	79.1	78.5
Octanal	2.8	2.2	1.9	5.5	4.9
Nonanal	18.8	12.6	12.6	19.0	18.9

The matrix is a partially hydrolyzed and pregelatinized rice flour. The aldehydes mass are expressed in ng/g for 40 g of sample.
Sample A : matrix aged during 16 weeks at room temperature.
Sample B : matrix aged during 2 weeks at 55°C.
Sample C : matrix with food additives (E170 & E340ii) aged during 16 weeks at room temperature.

Conclusions

The TD-GC/MS method was suitable for a qualitative and quantitative evaluation of rice flour volatile compounds. Among them, three aldehydes were considered as easily changing with rice evolution. For the three studied samples, level of Hexanal was always the highest. Conditions of storage and conditioning seem to have an influence mostly on the Hexanal level.