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Odour-impact compounds of an odour representative HS-SPME-extract of a red berries yoghurt drink: a D-GC-O and GC-MS/FID-O study

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Introduction

Headspace-solid-phase microextraction (HS-SPME) sampling followed by gas chromatography (GC) separation is widely used for the analysis of odorant compounds in dairy products (1, 2). However, depending on the fibre coating, extracts with varying odorant properties might result. Therefore, when analysing a product's odour and prior to GC-Olfactometry (GC-O) studies, it is highly useful to study extract representativeness. Direct-GC-O (D-GC-O) evaluates an extract's global odour at the sniffing port without chromatographical separation and a comparison to the original sample's odour is made. Hence, this technique is suitable for HS-techniques where no physical extracts are obtained (3−7).

Objectives

- determination of the main odour-impact compounds of an odour representative red berries yoghurt drink HS-SPME-extract by GC-MS/FID-O
- investigation of the odour representativeness of HS-SPME-extracts obtained with different fibre coatings by D-GC-O prior to GC-MS-O studies

Extract odour representativeness by D-GC-O

- evaluation of the HS-SPME-extracts' global odours compared to the original sample by rating their similarity with the original product on a scale from 0 (different) to 3 (identical) and their odour intensity (1 = weak, 2 = medium, 3 = strong) with a trained panel (*n*= 7)

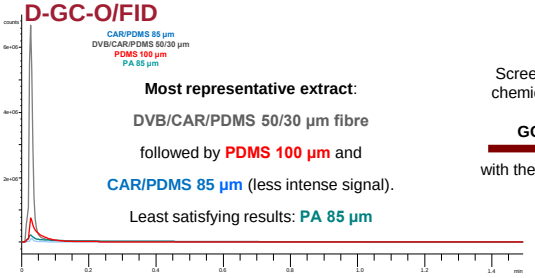
Odour-impact compounds by GC-MS/FID-O

- GC-MS/FID-O-analyses (trained panel *n*= 8) on two stationary phases (DB-5ms/VF-WAXms)
- odour-impact of constituents was evaluated by multiplying the detection frequency (*DF*, percentage of panellists perceiving an odour) with the mean intensity on a scale from 1 to 3
- use of methyl octanoate as internal standard (IS) for FID data processing

Sample extraction conditions for all analyses

- commercial red berries yoghurt drink
- 10 g in 20 mL SPME-HS-vial
- sample conditioning: 10 min at 40°C, $t_{adsorption}$= 5 min
- fibres: **CAR/PDMS 85 µm**, **DVB/CAR/PDMS 50/30 µm**, **PDMS 100 µm**, **Polyacrylate (PA) 85 µm**

Odour representativeness by D-GC-O and GC-MS-Olfactometry studies



Screening of volatile chemical and odorant profile by **GC-MS/FID-O** with the best three fibres

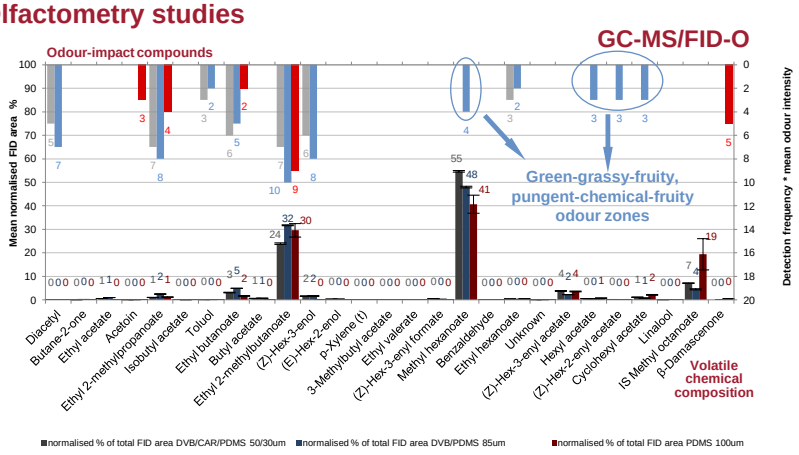


Fig. 2 Chemical composition (down) and odour-impact compounds (up) of HS-SPME red berries yoghurt drink headspace extracted with the three best 1cm-SPME fibres. Analyses (*n* = 2) were done on a DB-5ms column (panel *n*= 4). Only odorant compounds perceived by ≥ 50 % of the panel are listed.

Detailed study of the odour-impact compounds in the most representative extract: DVB/CAR/PDMS 50/30 µm fibre

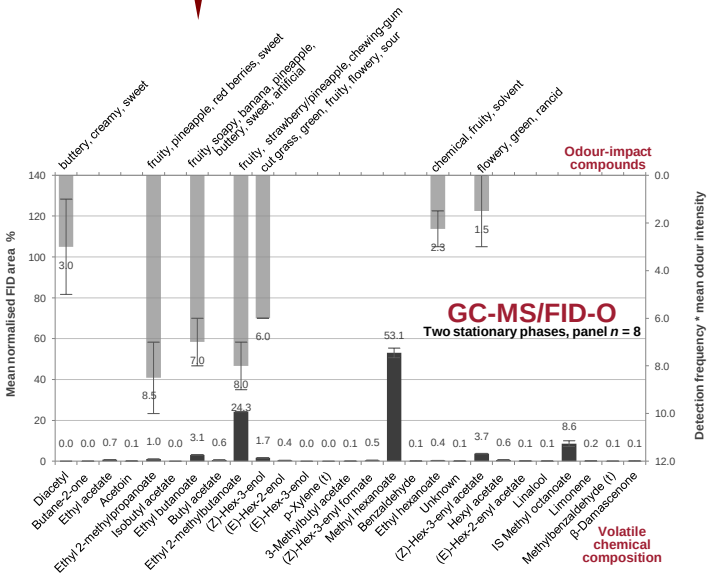


Fig. 3 Overlay of volatile chemical composition (down) and mean GC-O-profile (up) of HS-SPME red berries yoghurt drink extract obtained with a 1cm DVB/CAR/PDMS 50/30 µm fibre. Analyses were done on two different stationary phases (DB-5ms/VF-WAXms and four panellists smelled the sample on each column (panel total *n*= 8). Only odorant compounds perceived by either 75 % of the panel on one column or by 50 % on both columns are listed.

Conclusion

Seven odour-active zones were perceived by the panellists during GC-O-analyses at reliable detection frequencies (*DF ≥ 50 %*) with the DVB/CAR/PDMS 50/30 µm fibre, five with the PDMS 100 µm fibre, and eleven with the CAR/PDMS 85 µm fibre. Fruity notes were dominant. Four mainly green-grassy and chemical-fruity odour zones were perceived with the CAR/PDMS 85 µm fibre only. This result, together with the fact that the overall D-GC/FID signal intensity was lower for this fibre, might explain why this fibre gave less representative extracts and demonstrates the importance of testing odour-representativeness prior to extensive GC-O-work.

In-depth GC-O-analyses were done on a representative extract obtained with the DVB/CAR/PDMS 50/30 µm fibre (panel *n*= 8). The main odour-active compounds were ethyl 2-methylpropanoate (fruity, red berries, pineapple; present at ~1 %), ethyl 2-methylbutanoate (fruity, strawberry, pineapple; ~24 %), ethyl butanoate (fruity, banana, pineapple, soapy; ~3 %), and (Z)-hex-3-enol (grassy, fruity; ~2 %). Methyl hexanoate (~54 %) and the added IS methyl octanoate were not perceived, most likely due to their relatively high odour thresholds.

Literature 1. Arthur C.L.; Pawliszyn J. *Anal. Chem.* **1990**, *62*, 2145−2148; 2. Lehotay S.J.; Hajslova J. *Trends Anal. Chem.*, **2002**, *21*, 686−697; 3. Lecanu L. *et al. J. Agric. Food Chem.* **2002**, *50*, 3810−3817; 4. Rega B. *et al. J. Agric. Food Chem.* **2003**, *51*, 7092−7099; 5. Landy P. *et al. J. Agric. Food Chem.* **2004**, *52*, 2326−2334; Dury-Brun C. *et al. Flavour Fragrance J.* **2007**, *22*, 255−264; Breme K. *et al. Conference proceedings of the 9th Wartburg Symposium on Flavor Chemistry and Biology* **2011**, in press.; 6. Leffingwell *et al.* Services for the perfume, flavor, food, and beverage industry – odor thresholds, 01/09/2011, http://www.leffingwell.com/odorthre.htm

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