Volatile compounds contributing to the odour of oats

Sari A. Mustonen, OSKAR LAAKSONEN and Kaisa M. Linderborg

Food Chemistry and Food Development, Department of Life Technologies, University of Turku, Finland
oskar.laaksonen@utu.fi

Abstract

Oats are increasingly popular due to their healthiness, and the number of new different types of oat products on the market is constantly increasing. Oats have higher content of lipids compared to many other grains and therefore their quality and volatile compound profile is susceptible to changes. In this study, selected oat samples were investigated using HS-SPME-GC-O panel and trained sensory panel in order to identify the compounds contributing to the odour characteristics. GC-O panel was trained to describe odours and to evaluate odour intensities of oat samples as flour-water mixtures. The odour and flavour characteristics of the same oat samples were characterised using a sensory panel using generic descriptive analysis. The GC-O panel detected 30 odour-active compounds. The most often described compounds were aldehydes, such as hexanal described as 'green' and 'grassy', or 3-methylbutanal described as 'chemical' and 'pungent'. At the same time, little differences were observed in 'green odour' by the sensory panel, whereas more differences were observed in bitter taste and odour and flavour intensities.

Keywords: oat, volatile compounds, odour, flavour, sensory analysis

Introduction

The importance of oats is increasing globally due to the need for a shift to plant-based diet and public health concerns. A variety of oat products and fractions are available for use as such and as oat-containing foods, and the number of different types of oat products on the market is constantly increasing. Due to the extensive exploitation of oats in food industry, there are interests and concerns among oat producers, industry and researchers to better understand the factors affecting the quality of oats. Oats have higher content of lipids compared to many other grains and therefore their characteristics including the quantity and quality of volatile compounds is susceptible to changes, and certainly affected by processing [1,2]. Many of the compounds formed during processing are derived from oxidation of oat lipids [1-3]. At the same time, the odour of unprocessed oats is mild, and the typical odour characteristics are formed in various processes, such as heat treatment during milling [3].

The study was conducted as part of a larger OatHow research consortium in Finland aiming to investigate and define quality factors of oats. In this study, selected oat flour samples of Finnish origin were investigated using HS-SPME-GC-O panel and trained sensory panel in order to identify the compounds contributing to the odour characteristics of oats. The selected samples originated from a single crop year (either 2017 or 2018 and a known cultivar, and were industrially dehulled, heat-treated and milled from flakes.

Experimental

Gas chromatography olfactometry (GC-O)

GC-O analyses were performed with a Hewlett-Packard HP6890 Series GC system (Agilent Technologies Inc., CA, USA) coupled with a flame ion detector (FID) and an olfactometry port (ODP-1, Gerstel GmbH & Co. KG, Germany). A portion of 12 g of sample was mixed with MQ water (1:2.5, w/w) and placed in a 50 mL Erlenmeyer flask with 10% NaH_2PO_4. The sample was equilibrated and stirred thoroughly using a magnetic stirrer at 50 °C for 10 min. The SPME fibre (DVB/CAR/PDMS, 2 cm; Supelco, USA) was exposed to the headspace of the sample vial for 45 min at 50 °C. A medium polar capillary column (DB-624, 60 m×0.25 mm×1.4 µm, Agilent Technologies Inc., USA) was used to separate the compounds. The oven temperature-program was 40 °C held for 6 min, increased at 25 °C/min to 100 °C and then 7 °C/min to 220 °C and held 10 min at the final temperature. The injector temperature was set to 240 °C, and splitless injection was used. Helium was used as a carrier gas with linear velocity of 38 cm s^{-1}. Temperature of FID was set to 290 °C, and sampling rate to 20 Hz. Identification of compounds was performed using the retention indices and standard compounds. Chromatographic data were collected using GC ChemStation software (Rev. A.09.01, Agilent Technologies Inc., CA, USA) and olfactometric data using mp3DirectCut freeware (version 2.22).

The panel (n=5) was first trained to describe odours and to evaluate odour intensities (on scale 1-5; 1 = no odour, 2 = barely detectable, 3 = detectable, barely recognisable, 4 = recognisable, 5 = recognisable, strong) with odour bottles and then in GC-O with mixtures of standard compounds, and later with oat sample. The panel

DOI: 10.5281/zenodo.5913244

continued to evaluate four oat samples as flour-water mixtures (40 weight-% flour / 60 weight-% water) in duplicate.

Sensory evaluation

The odour and flavour characteristics of the oat samples were characterised using a sensory panel (n=11) using generic descriptive analysis. Sensory attributes (four odour attributes: 'oat', 'roasty', 'sweet' and total intensity; five flavour attributes: 'green', 'oat', total intensity and bitter and sweet tastes) were evaluated on line scales (0-10) with a help of reference compounds in triplicate by the panel. Oat flour-water mixture samples were prepared as described above. Samples (ca. 2.5g) were presented in 4 cL transparent plastic cups with glass lids in randomised order. Data was collected using Compusense Cloud software version 21.0 (Compusense Inc., Guelph, Ontario, Canada) in controlled sensory laboratory conditions.

Statistical analysis

Principal component analysis (PCA) models were used to investigate correlations between oat samples and perceived compound intensities by the GC-O panel (as mean-centred and unit-variance scaled as X-data) or sensory attributes (as mean-centred X-data). PCA models were created suing Unscrambler (version 11, Camo Inc., Norway). Rated intensities for samples in GC-O analyses were compared using oneway-ANOVA (SPSS version 27, SPSS Inc., Chicago IL).

Results and discussion

The GC-O panel (n=5 in duplicate) detected 30 potentially odour-active compounds from the oat flour-water mixture samples (Table 1). Eighteen compounds were detected at least five times or more often. The most often described compounds were aldehydes, for example hexanal (9) described as 'green' and 'grassy', or 3-methylbutanal (6) described as 'chemical' and 'pungent'. Hexanal, 1-octen-3-ol (with typical 'mushroom' descriptor) and 2,3-butanedione (commonly referred as diacetyl; with fatty, popcorn and sweet descriptors) were among the compounds rated as most intense on the scale. Aldehydes and ketones are among the typically detected compounds in oat samples (e.g. in review article by McGorrin [1]). Schuh and Schieberle (2004) reported a nonatrienal compound ((*E*,*E*,*Z*)-2,4,6-nonatrienal) being the key compound contributing to aroma of oats already in low concentrations. This compound was not detected in this study due to lack of standard. Potentially, it may be one of the unidentified compounds with number 25, 26 or 27. Many other compounds that were not detected in this study, have been reported in oats, especially compounds that are formed in further processing of the oats [1-3].

Only three compounds (7, 16, 20) differed statistically significantly in their intensities in oneway-ANOVA (Table 1). The intensity of compound 7 (2-methylbutanal with 'sweaty', 'fatty' and 'chemical' descriptors) was rated significantly lower in sample Oat3, compound 16 (unidentified) was lowest in sample Oat2 and compound 20 (nonanal with 'sweet' and 'fresh') was not detected in Oat4. At the same time, several compounds, such as 2-methylbutanal (7) with 'sweaty' and 'fatty' descriptors and the unidentified compound (25) with 'oat', 'roasty' and 'chemical', were observed differently among the oat samples in a PCA model in Figure 1.

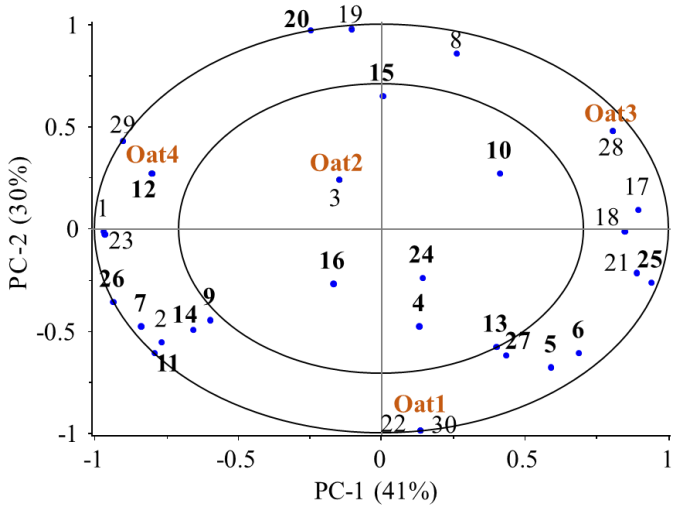

Figure 1: *PCA correlations loadings plot (PCs 1 vs 2) with four oat flour-water mixture samples (Oat1-4, brown font; included as dummy variables) and 30 compound variables from GC-O analysis (as average rated intensities on scale 1-5). Variable numbers and bolded variables refer to Table 1.*

Sample Oat1 was characterised by lacking nonanal (20) along the PC2, whereas the samples Oat3 and Oat 4 were separated on the first PC.

Table 1: Volatile compounds, their retention indices, odour descriptors and averaged rated intensities (on a scale 1-5 in duplicate) detected by the GC-O panel (n=5).

Compound number	RI (DB-624)	Compound	Descriptors by GC-O panel	Oat1	Oat2	Oat3	Oat4
1	<590	-	fusty, rancid, sweaty	3.8	3.3	3.0	3.3
2	<590	-	meat, rotten, pungent	3.0	3.0	1.0	3.2
3	591	-	-	1.0	2.0	1.0	1.0
4	**596**	**2-methylpropanal**	**solvent, fusty, sweet**	**2.9**	**3.4**	**3.0**	**3.3**
5	**634**	**2,3-butanedione**	**fatty, popcorn, sweet**	**3.6**	**3.5**	**3.9**	**4.1**
6	**696**	**3-methylbutanal**	**pungent, chemical, sweet**	**3.3**	**3.5**	**3.5**	**3.6**
7	**705**	**2-methylbutanal**	**sweaty, fatty, chemical**	**3.2^{ab}**	**3.0^{ab}**	**1.0^{a}**	**3.1^{b}**
8	**738**	**Pentanal**	**green, rancid, fatty**	**2.6**	**2.5**	**2.7**	**2.4**
9	**842**	**Hexanal**	**green, grass**	**4.2**	**4.4**	**3.9**	**4.3**
10	**876**	**-**	**sweet, flowery, fruity**	**2.6**	**3.3**	**3.0**	**2.8**
11	**909**	**Furfural**	**oat, flour, roasty**	**3.2**	**3.0**	**2.7**	**3.2**
12	**935**	**2-heptanone**	**mushroom, flour**	**2.0**	**1.0**	**1.0**	**1.0**
13	**948**	**Heptanal**	**mushroom, flour, fusty**	**3.1**	**2.9**	**3.2**	**3.3**
14	**980**	**-**	**mushroom, potato, fusty**	**3.6**	**3.1**	**3.1**	**3.5**
15	**1026**	**1-octen-3-ol**	**mushroom**	**4.1**	**3.9**	**4.1**	**3.9**
16	**1036**	**-**	**pungent, fatty, spicy, roasty**	**4.4^{b}**	**2.8^{a}**	**4.2^{ab}**	**4.3^{ab}**
17	1051	Octanal	green, fresh	1.0	3.0	3.5	2.5
18	1127	(*E*)-2-octenal	sweaty, rancid, roasty	1.0	3.3	3.5	2.8
19	1144	-	sweet, chemical, dusty	2.8	2.3	2.8	1.0
20	**1153**	**Nonanal**	**sweet, fresh**	**2.5^{ab}**	**2.3^{b}**	**2.3^{b}**	**1.0^{a}**
21	1162	-	hay, mould, medicinal	1.0	1.0	2.7	2.2
22	1179	-	sweet, roasty, toffee	1.0	1.0	1.0	2.5
23	1213	-	green, citrus	3.0	2.7	1.0	2.0
24	**1232**	**(*E*)-2-nonenal**	**oat, flour, green, fresh**	**3.4**	**3.1**	**3.4**	**3.4**
25	**1247**	**-**	**oat, roasty, chemical**	**2.8**	**3.3**	**3.6**	**3.5**
26	**1302**	**-**	**oat, roasty**	**3.5**	**3.2**	**2.8**	**3.3**
27	**1368**	**-**	**fusty, spoiled, oat**	**2.9**	**2.6**	**3.1**	**3.3**
28	1415	-	flour, dusty	1.0	1.0	2.3	1.0
29	1443	-	flour, fresh, roasty	3.0	2.3	1.0	1.0
30	1471	-	chocolate, vanilla	1.0	1.0	1.0	3.0

Compounds with bold font detected in at least 50% of GC-O panel evaluations. Statistically significant differences between rated intensities is based on oneway-ANOVA and Tukey's HSD test and shown with letters a-b.

Only little differences were observed between samples by the sensory panel in odour characteristics, whereas more differences were observed in bitter taste and total odour and flavour intensities. Sample Oat4 had the most intense odour, flavour and bitterness based on the PCA model in Figure 2. At the same time, Oat2 had milder odour and flavour with some positive correlation with 'oat odour' along the first PC. Sample Oat3 correlated with 'oat flavour' along the PC2. This sample also correlated with compound 25 (with descriptor 'oat') in the Figure 1.

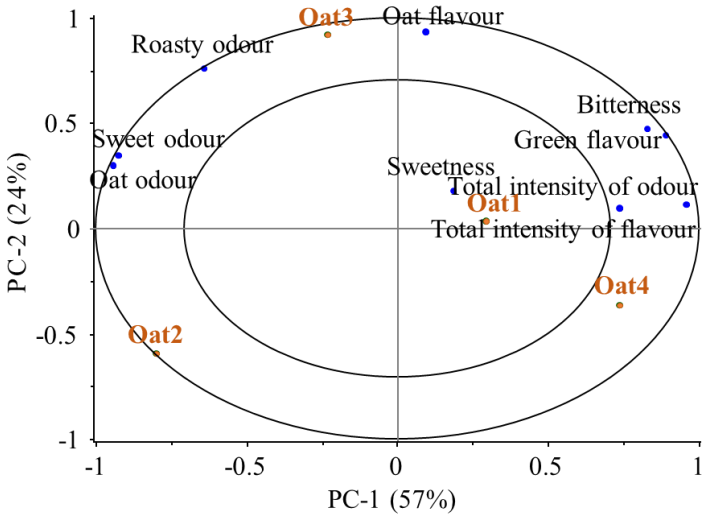

Figure 2*: PCA correlations loadings plot (PCs 1 vs 2) with four oat flour-water mixture samples (Oat1-4, brown font; included as dummy variables) and nine sensory variables from the generic descriptive analysis (as mean rated intensities on scale 0-10).*

Conclusion

Oat flour samples in this study had generally mild odour and flavour properties, and its odour was contributed by multiple volatile compounds. Only a part of the compounds described in literature, primarily aldehydes, were detected in this study. Oat flour samples selected to this study differed from one another in terms of the perceived odour as flour-water mixtures, especially those described as “sweaty”, “fatty” or with bitter taste. In subsequent studies, more oat samples with varying origins are investigated and the odour-active compounds will be studied in detail with GC-MS analyses. Additionally, a storage test of these flours will be carried out, as well as, certain concept products will be prepared from the flours in order to further investigate the suitability of different oat flours for different end-products.

References

1. McGorrin, R.J. Key aroma compounds in oats and oat cereals. J Agric Food Chem. 2019;67:13778−13789.
2. Schuh,C., Schieberle, P. Characterization of (*E,E,Z*)-2,4,6-nonatrienal as a character impact aroma compound of oat flakes. J Agric Food Chem. 2005;53:8699–8705.
3. Heiniö, R.-L.; Kaukovirta-Norja, A.; Poutanen, K. Flavor in processing new oat foods. Cereal Foods World 2011;56(1):21–26.