An analysis of the volatile flavour compounds in a soft raw goat milk cheese

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The major components of the musty cow rind cheeses were identified in a soft raw goat milk cheese as heptan-2-one, nonan-2-one, their corresponding secondary alcohols, some esters and sulfur compounds. Their production was associated with the manufacturing process and its influence on the microbial activity. However a specificity in goat cheese compounds was displayed concerning in particular limonene and some ketones, alcohols and aldehydes.

Introduction
The cheese studied in this work is a soft mold-ripened cheese produced in the Poitou-Charentes region of France from raw goat milk. The aroma composition of soft and musty rind cheeses made with cow milk, as Camembert, has received a great deal of investigation (Molimard and Spinnler, 1996). By contrast no data have been published on the volatile flavour compounds of the studied cheese or any other French goat milk cheese. Only a traditional Spanish goat cheese have been studied (Vidal-Aragon et al., 1994). In the present work, the volatile compounds of our goat cheese were investigated during ripening.

Materials and methods
Samples
The tradionnal-making cheeses were supplied by Lescure-Bougon factory (Bougon, 79800 France). Each cheese was vacuum-packaged in plastic bags, frozen and stored at -80°C until analysis. Before analysis, the frozen samples were ground to powdered form and thoroughly mixed to obtain representative samples. All the preparation steps were carried out at 4°C.

Purge and trap extraction
A purge and trap injector (model 3000, Tekmar, U.S.) was used. One gram of sample was transferred into a 25 ml purge bottle. The bottle was sealed air-tight on the purge and trap injector, and pressurized with helium. The sample bottle was preheated for 10 min and then maintained during the purge time at 30°C in a water bath. The sample bottle was purged with helium at 50 ml/min for 30 min in order to isolate headspace volatile compounds, which were adsorbed on a 24 cm Tenax trap column (Tekmar, U.S.). Then, the volatiles were thermally desorbed at 200°C for 5 min and were concentrated in a cold trap (MFA 815, Fisons Instruments, France) maintained at -150°C with liquid nitrogen. The volatile compounds condensed in the cryofocusing unit were automatically injected into the GC column by heating the cryofocusing unit at 200°C.

Gas chromatography
Separation of volatiles was performed using a GC 8000 (Fisons Instruments, France) equipped with a mid-polar capillary column (DB 1701, 30 m x 0.32 mm, 1 μm film thickness, J&W Scientific, U.S.) and a flame ionisation detector (FID). The program of oven temperature was: 40°C for 10 min, increase at 2°C/min to 100°C and then at 20°C/min to 220°C, finally 220°C for 5 min. Helium carrier gas was used at a flow rate of 2.4 ml/min. All analyses were done in triplicate. The technique reproducibility was assessed in analysing five times 43-days-old cheese.

Quantitative analysis
Standard amounts (five points, in duplicate) of pure compounds from stock solutions in propan-2-ol (except propanal, acetone, hexane, propan-2-ol in propanol) were injected by the split injector of GC. Linear regression curves were calculated for each compound. The quantity of the compounds extracted from cheese was expressed by g of cheese.
Table 1 | Volatile organic compounds identified in soft mold-ripened goat cheese

<table>
<thead>
<tr>
<th>PN</th>
<th>RT</th>
<th>CV</th>
<th>Quantity versus ripening days</th>
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<tbody>
<tr>
<td>2</td>
<td>8</td>
<td>15</td>
<td>22</td>
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</tbody>
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ketones

- acetone
  - 7 3.6 GC, MS 10.5 12.3 52.3 38.1 46.4 43.7 37.4 58.7 80.8
- butan-2-one
  - 17 6.7 GC, MS 10.0 45.1 27.2 7.2 8.4 7.7 1.1 7.9 9.5
- pentan-2-one
  - 25 13.0 GC, MS 12.9 21.3 40.4 12.1 37.8 30.0 28.1 26.3
- hexan-3-one
  - 35 21.6 GC, MS 2.7 – 2.2 2.4 3.9 1.2 0.1 0.2
- hexan-2-one
  - 36 22.6 GC, MS 12.2 7.3 5.9 11.2 13.2 21.7 10.0 8.9
- heptan-2-one
  - 43 29.8 GC, MS 11.0 – – – – – 9.2 15.0
- heptan-2-one
  - 44 32.5 GC, MS 10.4 41.6 137.2 221.1 253.0 289.7 320.0 435.2

3-methylheptan-2-one

- 48 36.7 GC, MS 11.6 – – 1.3 1.2 1.5 2.3 4.2

- 50 40.5 GC, MS 12.3 – – 1.7 2.0 1.5 2.1 9.3
- 57 41.3 GC, MS 11.3 1.4 10.5 1.9 3.2 28.7 26.8 24.8
- nonan-4-one
  - 59 43.6 MS 7.5 – – – 3.4 3.5 4.3 4.3
- nonan-2-one
  - 60 44.3 GC, MS 8.0 17.2 123.8 148.6 211.3 297.6 320.0 407.4

alcohols

- ethanol
  - 5 3.2 GC, MS 8.9 1971.9 1472.2 1183.9 1194.7 997.3 598.2 441.2
- propan-2-ol
  - 9 3.9 GC, MS 9.4 81.5 26.9 153.9 150.8 27.9 64.8 100.8
- 2-methylpropan-2-ol
  - 11 4.6 GC, MS 10.7 – – 0.7 1.0 0.8 0.3
- propanol
  - 14 6.1 GC, MS 12.8 7.2 3.9 8.4 6.4 3.4 3.0 5.6
- 2-methylpropanol
  - 22 10.1 GC, MS 10.1 104.7 64.9 50.4 49.3 21.7 10.0 8.9
- methoxypropan-2-ol
  - 24 12.2 GC, MS 10.1 104.7 64.9 50.4 49.3 21.7 10.0 8.9
- butanol
  - 26 13.2 GC, MS 12.5 – – 3.8 17.1 16.2 2.6 8.2 6.8
- pentan-2-ol
  - 30 40.5 GC, MS 12.9 21.3 40.4 12.1 37.8 30.0 28.1 26.3
- 3-methylbutanol
  - 44 20.5 GC, MS 10.4 41.6 137.2 221.1 253.0 289.7 320.0 435.2
- 3-methylbutanol
  - 48 36.7 GC, MS 11.6 – – 1.3 1.2 1.5 2.3 4.2
- octan-2-ol
  - 50 40.5 GC, MS 12.3 – – 1.7 2.0 1.5 2.1 9.3
- nonan-2-ol
  - 59 43.6 MS 7.5 – – – 3.4 3.5 4.3 4.3
- nonan-2-ol
  - 60 44.3 GC, MS 8.0 17.2 123.8 148.6 211.3 297.6 320.0 407.4

aldehydes

- acetaldehyde
  - 2 2.2 GC, MS 8.3 9.5 24.4 20.4 17.5 48.5 89.9 105.7
- propanal
  - 6 3.4 GC, MS 12.8 – – – – 1.5 4.9 4.4
- 2-methylpropanal
  - 12 4.7 GC, MS 12.6 3.1 4.0 3.6 6.6 4.2 3.2 8.3
- 3-methylbutanal
  - 21 9.7 GC, MS 8.2 2.0 61.8 25.4 26.2 24.0 27.8 119.5
- hexanal
  - 37 23.0 GC, MS 10.4 24.5 24.7 9.9 12.3 1.8 8.8 11.5
- octanal
  - 58 41.5 GC, MS 9.0 2.1 3.3 1.8 3.6 2.6 3.6 3.0

esters

- ethyl acetate
  - 15 6.3 GC, MS 11.3 196.9 47.1 2.0 2.7 9.1 21.4 4.0
- ethyl butyrate
  - 34 20.8 GC, MS 10.3 – – 63.2 10.0 7.6 7.3 0.7 1.1
- ethyl hexanoate
  - 53 39.3 GC, MS 9.7 – 4.8 7.8 5.3 1.1 1.2 0.5

sulfur compounds

- dimethylsulfide
  - 60 15.7 MS 7.4 1.8 0.3 0.2 0.2 3.6 6.1 1.6
- dimethylsulfoxide
  - 49 37.5 MS – – – – – –

hydrocarbons

- pentane
  - 3 2.4 MS 6.5 – – 4.3 2.9 3.3 1.5 1.6 2.0
- hexane
  - 8 3.7 GC, MS 9.7 – – – 3.3 2.3 6.4 7.6
- 2-methylhexane
  - 13 5.6 GC, MS 7.8 – – – – – – 0.6 0.8 1.9
- methylcyclohexane
  - 20 9.2 GC, MS 6.4 – – – – – – 0.6 0.8 1.9
- toluene
  - 31 16.4 GC, MS 11.5 20.4 21.9 14.6 19.3 14.4 14.1 15.9
- xylene
  - 38 24.6 GC, MS 9.0 – – 3.2 – 2.8 2.8 1.4 1.0
- ethylbenzene
  - 40 25.4 GC, MS 9.2 4.6 3.7 2.8 4.0 6.9 5.2 5.8
- p-xylene
  - 41 26.3 GC, MS 9.1 11.3 4.5 7.9 10.5 19.6 19.1 18.1
- methylstyrene
  - 50 38.7 GC, MS 10.4 – 2.5 2.6 3.4 2.4 1.3 3.6
- limonene
  - 52 38.9 GC, MS 9.3 4.1 58.5 75.3 53.9 54.3 58.7 55.7

PN, peak numbering gives the order of elution; RT, column retention time (min); GC, gas chromatographic retention data compared with those of pure compounds and increase of the peak area by adding pure compounds to cheese sample; MS, mass spectral data compared with those of library compounds; CV, coefficient of variation obtained for five analyses of 43-days-old cheese (peak area standard deviation/mean, %); (a) in ng/g of cheese; (b) 3-methylheptan-2-one quantity was determined with octan-3-one regression curve; (c) nonan-4-one quantity was determined with nonan-2-one regression curve; (d) ethanol quantity, which caused FID saturation, was estimated on the reconstructed total ion chromatogram in MS; (–) not detectable; (e) area <1000 μV·sec.