

Analysis of Aroma Components in Milk Using Smart Aroma Database™

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User Benefits

- ◆ Approximately 500 aroma-related components are registered in the Smart Aroma Database, supporting efficient analysis of aroma components.
- ◆ Concentration of aroma components by SPME Arrow enables satisfactory analysis of milk in spite of its low concentration of aroma components.

Introduction

The aroma components of milk are one of the important factors that determine its deliciousness, but the components differ greatly depending on the milk producing area, animal feed, and milk sterilization and storage methods. However, due to the extremely low concentration of aroma components in milk, a comprehensive analysis of a large number of aroma components is considered difficult. In this experiment, six types of milk were sampled and their aroma components were concentrated by the solid phase microextraction method (SPME Arrow). The samples were then analyzed by a GC/MS using the Shimadzu aroma component database Smart Aroma Database, and a comparative study of the milk samples was conducted.

Analysis Method and Samples

Table 1 shows the analysis conditions. An SPME Arrow was used in the solid phase microextraction. As shown in Fig. 1, an SPME Arrow holds approximately 5 to 20 times as much adsorbent as a conventional SPME fiber, enabling analysis with high sensitivity, and due to its thick, sturdy structure, the SPME Arrow fiber also boasts excellent durability in comparison with conventional SPME fibers. Here, six types of milk with different producing areas and sterilization methods were sampled, and the samples were sealed in 3 mL vial. After heating at 40 °C for 30 min, the solid phase microextraction was performed and an analysis of the aroma components was conducted using the analysis conditions of the Smart Aroma Database. (For the analysis flow of the Smart Aroma Database, please refer to Application News 01-00316)

Table 1 Instrument Configuration and Analysis Conditions

Instrument	
GCMS	: GCMS-QP2020 NX
Database	: Smart Aroma Database
Autosampler	: AOC-6000 Plus
Column	: InertCap® Pure Wax (Length 30 m, 0.25 mm I.D., df = 0.25 µm)
SPME Arrow	
SPME Arrow	: 1.1 mm O.D. DVB/Carbon WR/PDMS, 120 µm
Conditioning Temp.	: 270 °C
Pre Conditioning Time	: 10 min
Incubation Temp.	: 40 °C
Incubation Time	: 5 min
Stirrer Speed	: 250 rpm
Agitator Speed	: 250 rpm
Sample Extract Time	: 30 min
Sample Desorb Time	: 2 min (250 °C: GC Injection Temp.)
GC Conditions	
Injection Temp.	: 250 °C
Injection mode	: Splitless (sampling time: 1 min)
Purge flow rate	: 3.0 mL/min
Control mode	: Pressure (83.5 kPa)
Column oven temperature	: 50 °C (5 min) → 10 °C/min → 250 °C (10 min)
MS Conditions	
Interface temperature	: 250 °C
Ion source temperature	: 200 °C
Ionization method	: EI
Acquisition mode	: Scan
Event time	: 0.3 s

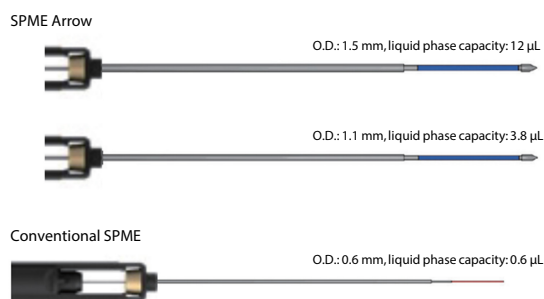


Fig. 1 Comparison of SPME Arrow and Conventional SPME Fiber

Analysis Results

Peak identification of aroma components registered in the Smart Aroma Database was carried out based on their registered retention time information, ion information, and mass spectrum information, and it was possible to identify 45 types of aroma components. Table 2 shows the identified compounds.

Table 2 Identified Aroma Components of Milk

Ethyl acetate	trans-2-Heptenal
Diacetyl	6-Methyl-5-hepten-2-one
alpha-Pinene	1-Hexanol
Dimethyl disulfide	Dimethyl trisulfide
Butyl acetate	2-Nonanone
2-Hexanone	3-Octanol
Hexanal	alpha,p-Dimethylstyrene
Isobutanol	Ethyl octanoate
Pyridine	1-Octen-3-ol
o-Xylene	1-Heptanol
2-Heptanone	Menthone
Limonene	2-Acetylfuran
2-Pentylfuran	Dimethyl sulfoxide
Ethyl hexanoate	1-Octanol
Styrene	2-Undecanone
1-Pentanol	Acetophenone
3-Octanone	Furfuryl alcohol
p-Cymene	Butylated hydroxytoluene
Hexyl acetate	delta-Octalactone
1,2,4-Trimethylbenzene	Caprylic acid
2-Octanone	delta-Decalactone
3-Heptanol	delta-Dodecalactone
2,6-Dimethylpyrazine	

Multivariate Analysis Results

A principal component analysis (PCA) of the analysis results of this experiment was carried out using the multivariate data analysis software SIMCA 17® (Infocom Corporation). Fig. 2 shows the score plot of the results. Two types of milk samples (milks ② and ⑥) were separated by a large distance on the score plot, indicating that their aroma characteristics differ greatly. Fig. 3 shows the loading plot. In order to investigate the details of milks ② and ⑥, which displayed different aroma characteristics, the components were checked from the loading plot. Table 3 shows some of the compounds that were detected in relatively large amounts in milks ② and ⑥. Figs. 4 and 5 show the comparison of the mass chromatograms of hexanal and ethyl hexanoate in milks ② and ⑥, respectively.

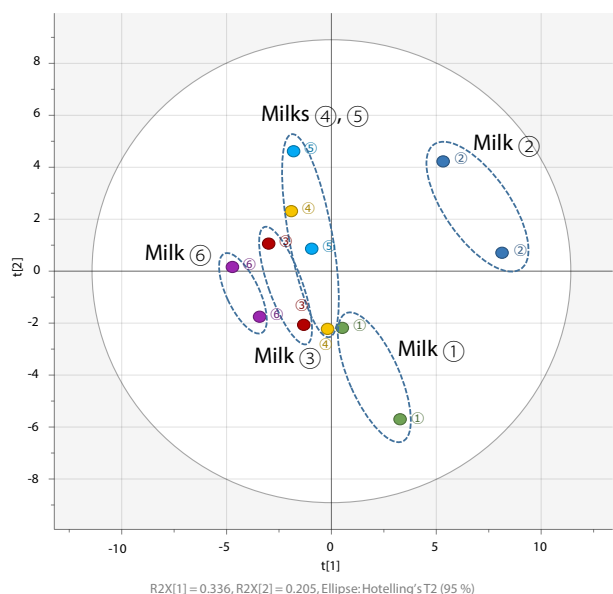


Fig. 2 Score Plot

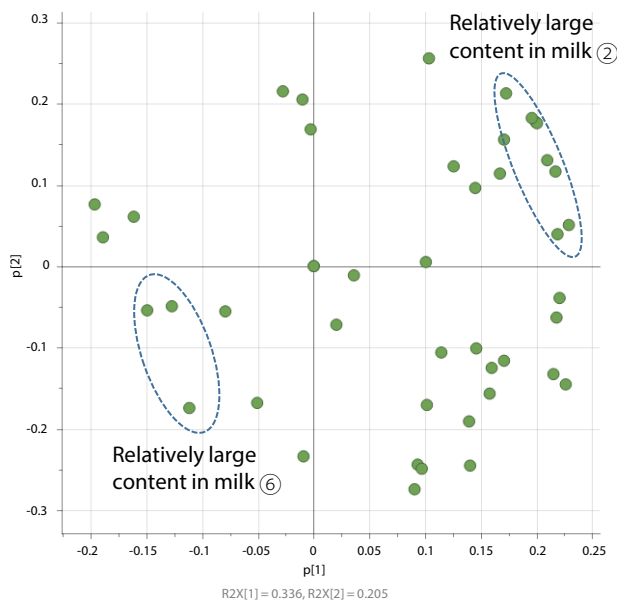


Fig. 3 Loading Plot

Table 3 Compounds Detected in Relatively Large Amounts in Milks ② and ⑥

Relatively large content in milk ②	Relatively large content in milk ⑥
1-Pentanol	Butyl acetate
1-Hexanol	Ethyl hexanoate
1-Heptanol	3-Heptanol
delta-Dodecalactone	
Hexanal	
Isobutanol	
2-Pentylfuran	
2-Hexanone	

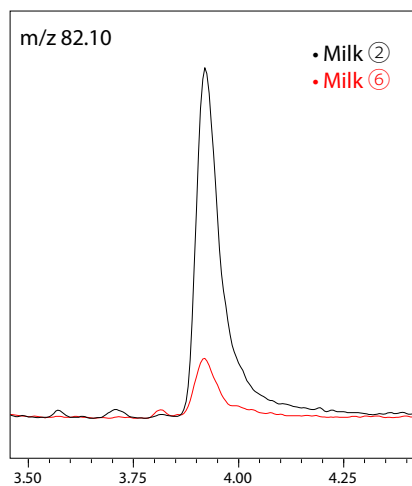


Fig. 4 Comparison of Mass Chromatograms of Hexanal in Milks ② and ⑥

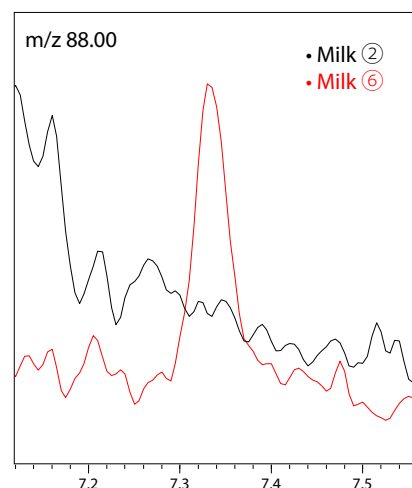


Fig. 5 Comparison of Mass Chromatograms of Ethyl Hexanoate in Milks ② and ⑥

Conclusion

The aroma components of six milk samples were concentrated by solid phase microextraction (SPME Arrow), and a GC/MS analysis was carried out using the Shimadzu aroma component database Smart Aroma Database. As a result, a comparative study of the aroma components of the various milk samples was possible.

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