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Multivariate approach to reveal relationships between sensory perception of cheeses and aroma profile obtained with different extraction methods

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ABSTRACT

A new and original statistical approach was used to compare the effectiveness of 4 different methods to analyse aroma compounds of seven different commercial semi-hard cheeses with regard to their orthonasal sensory perception. Four extraction methods were evaluated: Purge and Trap, Artificial Mouth, Solid-Phase Microextraction (SPME) and Solvent-Assisted Flavour Evaporation (SAFE). Among the headspace methods, Artificial Mouth gave the closest representation of the studied product space to the sensory perception one. The SAFE method was complementary to the dynamic headspace methods, as it was very efficient in extracting the heavy molecules but less efficient for extracting the most volatile compounds. SPME and Purge and Trap gave intermediate representations. At the product level, results indicate that carboxylic acids are present in similar amounts in the different commercial marks of semi-hard cheeses, that esters and aldehydes vary in small proportions and that the proportions of sulphur compounds, alcohols and ketones mainly explain the sensory differences between the commercial marks.

1. Introduction

Cheese aroma has been widely studied with many different analytical measurements (Le Quéré, 2004). However no single extraction method can account for the large variety of aroma compounds responsible for the sensory perception. The most applied extraction techniques are distillation (Barbieri et al., 1994; Larrayoz, Addis, Gauch, & Bosset, 2001; Poveda, Sanchez-Palomo, Perez-Coello, & Cabezas, 2008; Van Leuven, Van Caelenberg, & Dirinck, 2008), static headspace (Qian & Reineccius, 2003b), dynamic headspace (Bellesia et al., 2003; Fernadez-Garcia, Carbonell, Gaya, & Nunez, 2004; Mallia, Fernandez-Garcis, & Bosset, 2005; Zehentbauer & Reineccius, 2002), solid-phase microextraction (SPME) (Bellesia et al., 2003; Frank, Owen, & Patterson, 2004; Lecanu, Ducruet, Jouquand, Gratadoux, & Feigenbaum, 2002; Mallia et al., 2005; Peres, Viallon, & Berdague, 2001; Sadecka, Kolek, Pangallo, Valik, & Kuchta, 2014), stirbar sorption extraction (Panseri et al., 2008) and vacuum distillation (Lecanu et al., 2002). The headspace techniques are known to be efficient when extracting the most volatile compounds, such as small ketones and sulphur compounds. The distillation and solvent extractions are most efficient when studying the heavier, more hydrophobic molecules, such as free fatty acids and lactones. However, one disadvantage of the distillation method is the formation of artefact compounds that may be falsely associated with the natural cheese flavour (Le Quéré, 2004).

The main purpose of analysing cheese aroma is to find odour-active compounds that are responsible for the perceived flavour. Thus, an extraction method must be carefully chosen so that the identified aroma compounds are those that are transported to the olfactory epithelium by the orthonasal or retronasal route. Theoretically, distillation and solvent extraction techniques cannot be adapted because they break the matrix effect. Headspace methods allow the detection of highly volatile compounds, and they take the dynamic release from the matrix into account. Moreover, Artificial Mouth systems have been developed and optimized to simulate flavour release in oral vapour while consuming food products (van Ruth & Roozen, 2000).

Evaluation of the representativeness of an extract has been the subject of different developments in function of the extraction method and the product. The similarity between the real food product and the extract can be evaluated directly as in the case of beer extracted by different resins eluted with water (Abbott, Etiévant, Langlois, Lesschaeve, & Issanchou, 1993) or wine extracted by resin, demixion or solvent (Priser, Etiévant, Nicklaus, & Brun, 1997). Solvent extracts can be evaluated after placing a drop on a perfume sampling (Acena, Vera, Guasch, Busto, & Mestres, 2010) or reincorporated into an odourless matrix

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Table 1a
Sulphur compounds and alcohols identified in the 7 cheeses by mass spectrometry (MS) and linear retention index on DB-WAX column and their relative amounts extracted by SPME and SAFE (peak area relative to total peak areas, mean of 3 replicates).

		LRI			SPME							SAFE						
Molecule	logP[a]	exp[b]	lit[c]	Ident[d]	C1	C2	C3	C4	C5	C6	C7	C1	C2	C3	C4	C5	C6	C7
Sulphur compounds																		
Methanethiol	1.92	672	635	MS, LRI	0.00	0.00	0.12	0.33	0.00	0.24	0.24							
Dimethyl sulfide	0.89	751	745	MS, LRI, SI	0.71	0.76	0.46	0.37	0.10	0.32	0.36							
Dimethyl disulfide	1.77	1101	1075	MS, LRI, SI	0.38	0.84	2.06	4.93	0.36	3.49	6.48	0.06	0.28	0.00	0.55	0.00	0.23	0.28
Dimethyl trisulfide	2.93	1430	1383	MS, LRI	0.00	0.00	0.01	0.12	0.02	0.13	0.21	0.01	0.00	0.00	0.00	0.00	0.32	0.00
Sulfinylbis(methane)	−1.35	1575	1553	MS, LRI, SI	0.29	0.32	0.23	0.27	0.05	0.15	0.09	0.18	0.00	0.21	0.00	0.00	0.00	0.00
Dimethylsulphone	−1.41	1895	1833	MS, LRI								0.00	0.40	0.00	0.43	0.26	0.26	0.19
s-Methylthioacetate	0.73	1074	1056	MS, LRI, SI	0.00	0.00	0.00	0.00	0.00	0.00	0.35							
2-Methyltetra-hydrothiophen-3-one	0.2	1577	1538	MS, LRI								0.00	0.39	0.13	0.51	0.00	0.30	0.48
Alcohols																		
Propan-2-ol	0.05	934	975	MS, LRI	1.23	0.95	0.31	2.34	2.65	1.58	1.63							
Butan-2-ol	0.61	1039	1032	MS, LRI, SI	1.87	0.00	0.00	1.50	0.00	0.83	2.77	0.00	0.00	0.00	0.00	0.00	0.00	0.42
Propan-1-ol	0.25	1055	1052	MS, LRI, SI	0.99	0.00	0.00	0.00	0.00	0.00	0.14							
Methylpropan-2-ol	0.4	1116	1097	MS, LRI, SI	0.40	0.19	0.14	1.76	0.20	0.36	0.49	0.00	0.00	0.00	0.21	0.00	0.25	0.16
Pentan-2-ol	1.22	1143	1142	MS, LRI, SI	0.00	0.00	0.00	0.00	0.10	0.00	0.09							
1-Methoxypropan-2-ol	−0.44	1162	1160	MS, LRI, SI	0.19	0.47	0.00	0.00	0.16	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
1-Ethoxypropan-2-ol	0	1191	NA	MS	0.27	0.24	0.93	0.70	0.00	0.11	0.08	0.25	0.16	0.19	0.52	0.00	0.20	0.15
Butan-1-ol	0.88	1179	1152	MS, LRI, SI														
2-Methylbutan-1-ol	1.22	1233	1212	MS, LRI, SI														
3-Methylbutan-1-ol	1.22	1227	1215	MS, LRI, SI	6.10	0.00	0.15	8.23	6.91	3.34	3.24	2.24	0.11	4.64	5.19	8.85	6.22	5.93
3-Methyl-3-buten-1-ol	1.25	1271	1263	MS, LRI, SI	0.00	0.00	0.00	0.00	0.13	0.00	0.00							
2-Methylhexan-3-ol	2.17	1396	NA	MS														
2-Ethylhexan-1-ol	2.82	1500	1492	MS, LRI, SI								0.05	0.11	0.05	0.09	0.35	0.09	0.08
Octan-1-ol	2.88	1567	1561	MS, LRI, SI								0.14	0.16	0.06	0.01	0.34	0.15	0.19
Butan-1,2-diol	−0.29	1345	NA	MS								0.00	0.00	9.43	0.35	0.00	0.00	0.00
Propan-1,2-diol	−0.92	1597	1605	MS, LRI, SI	0.65	0.00	0.00	0.00	0.00	0.00	0.00	0.39	0.15	0.00	0.14	0.09	0.00	0.00
Butan-2,3-diol	−0.92	1548	1582	MS, LRI	1.50	2.68	2.06	2.31	0.98	1.83	1.03	1.45	0.99	0.41	0.96	0.35	0.61	0.22
Benzeneethanol	1.36	1913	1905	MS, LRI, SI	0.09	0.00	0.00	0.00	0.00	0.00	0.00	1.51	0.07	0.66	2.54	4.82	2.29	3.50
2-Butoxyethanol	0.83	1428	1441	MS, LRI, SI								0.00	0.00	0.04	0.06	0.00	0.00	0.00

[a] Hydrophobicity estimated by Epi Suite software (EPA's Office of Pollution Prevention Toxics and the Syracuse Research Corporation (SRC)): KOWWIN v1.67 estimate.
[b] Experimental linear retention index calculated by injection of a series of alkanes on a DB-WAX column.
[c] Linear retention index from literature.
[d] MS mass spectrum in agreement with literature; LRI in agreement with literature, SI: injection of standard in the same conditions.

resembling the food product (Etievant et al., 1993). However these modes of reincorporation are not possible for headspace extracts which are directly injected in a gas chromatograph. In that case the evaluation of the odour can be done by direct GC–olfactometry (Rega, Fournier, & Guichard, 2003) or after injection of this extract into a syringe (Poinot et al., 2007). These techniques were suitable for the optimisation of extraction parameters for one specific extraction method (Poinot et al., 2007; Qian, Nelson, & Bloomer, 2002; Qian & Reineccius, 2003a, 2003b, 2003c; Rega et al., 2003) because the same mode of presentation of the extracts was used. However it is more difficult to evaluate the representativeness of extracts derived from very different extraction methods because the extracts are not evaluated in the same type of matrix, e.g., gas phase for dynamic headspace methods and liquid phase for solvent extractions (Acena et al., 2010; Murat, Gourrat, Jerosch, & Cayot, 2012).

A different strategy to overcome this difficulty is proposed. In many studies the identification of aroma compounds has for aim to find the compounds which best explain the sensory differences between samples. Our study aimed to determine how a set of related food products are discriminated on the basis of sensory evaluation then on the basis of the relative quantity of aroma compounds extracted by different methods. Hence, our hypothesis was that the product discrimination pattern obtained with the extraction method that best represents the sensory product discrimination would contain the necessary information about the volatile compounds that influence the sensory perception. Principal component analysis has been successively applied to distinguish seven different cheeses in terms of their content in volatile compounds (Poveda et al., 2008), without any comparison with sensory data.

Finding relationships between different sets of variables has been the subject of various strategies. This is especially true in the field of foodstuffs characterisation performed by both physico-chemical measurements and sensory evaluations. Among the different strategies, the RV coefficient is based on a multidimensional approach to measure of similarity between p-dimensional and q-dimensional configurations of the same sample. It is based on the principle that two sets of variables are perfectly correlated if there exists an orthogonal transformation that makes the two sets coincide (Escoufier, 1973) and thus well adapted to the comparison of two factorial maps and detects the relationships between these configurations. It was proved to be a good way to define the proximity between variable clusters and compare different PCA configurations on six apricot cultivars (Schlich & Guichard, 1989). The transformation into a standardised RV (SRV) coefficient offers the possibility to test its significance and has been successfully applied to sensory analyses of wines (Josse, Pagès, & Husson, 2008).

The purpose of our study was to compare 4 different extraction methods in order to point out the differences in aroma compounds present in seven semi-hard cheeses which better explain the sensory perception. Solvent Assisted Flavour Evaporation is known to extract a great number of aroma compounds from different chemical classes present in the food. However methods based on the analysis of the vapour phase are often preferred such as Solid-Phase Microextraction and dynamic headspace followed by trapping of aroma compounds (Purge and Trap). In order to better reproduce the in-mouth conditions, we also tested an Artificial Mouth with the addition of artificial saliva. At the sensory level, the seven cheeses were evaluated by orthonasal sensory analysis through a free sorting methodology to obtain the perceptual differences between the cheeses in a multidimensional sensory space. This sensory discrimination was then compared to the multidimensional configuration of the cheeses obtained from each extraction method. The proximity between the multidimensional spaces was then assessed using the standardised RV coefficient. Because the