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Application of artificial neural network on mono- and sesquiterpenes compounds determined by headspace solid-phase microextraction–gas chromatography–mass spectrometry for the Piedmont ricotta cheese traceability

Zeppa Giuseppe*, Giordano Manuela, Bertolino Marta, Gerbi Vincenzo

University of Turin, Agricultural Faculty, Department of Exploitation and Protection of the Agricultural and Forestry Resources, Food Technology Sector, Via L. da Vinci 44, 10095 Grugliasco (TO), Italy

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Abstract

Mono- and sesquiterpenes were used for the traceability of a typical Piedmont (Italy) mountain ricotta cheese produced by nine mountain farms. For each farm a sample of ricotta cheese was collected every 7 days during mountain grazing and analysed using headspace solid-phase microextraction–gas chromatography–mass spectrometry (SPME–GC–MS). Obtained results showed the presence of about 20 monoterpenes (above all α-pinene, β-pinene, camphene, *p*-cymene, β-myrcene and limonene) and about 15 sesquiterpenes such as α-caryophyllene, α-copaene and 9-epi-caryophyllene. Despite a wide concentration variability due to the stages of plant development and the pastured area, there are not able differences between the ricotta cheeses analysed so it is possible with the artificial neural network (ANN) technique to distinguish between different mountain farms.

Keywords: Cheese; Monoterpenes; Sesquiterpenes; Solid-phase microextraction–gas chromatography–mass spectrometry; Headspace analysis; Artificial neural network

1. Introduction

Animal feeding is a very important factor in cheese characterisation due to its action on milk bacteria and milk compounds such as fats, proteins, flavours, bacteria and so on [1,2]. Several studies have highlighted that cheeses obtained by animals feeding on pasture with dicotyledons have more flavour than those obtained by animals feeding with hay or cereal or monocotyledons [3–5]. This difference is due to the presence in the dicotyledons of terpenes and over all mono- and sesquiterpenes. These compounds, secondary metabolites located in particular differentiated cells of trichomes, are known for their double properties: disinfectant and odorant for their odour described as 'fresh, herbaceous, resin, lemon and coniferous'. These molecules are present in many plants and their content is related to botanical species (Apiaceae and Fabaceae are richer than monocotyledons such as Poaceae) and to the plant's phenological phase [4]. Since dicotyledons are more abundant in the highlands than in the lowlands where the monocotyledons are more abundant, terpene compounds are more abundant in milk and cheeses produced in mountain farms. Dumont et al. [3] showed that sesquiterpenes are present only in the Beaufort cheese produced during summer when the cows graze in the highlands. Mariaca et al. [4] in Gruyère and Etivaz cheeses produced in highlands have identified 42 terpenes such as β-myrcene, linalool, limonene, α-phellandrene, α-terpinene, δ-3-carene, *p*-cymene, β-pinene, α-copaene and α-humulene also present in 13 grass family mostly dicotyledons (Apiaceae, Asteraceae, Fabaceae and Rosaceae). Bugaud et al. [6] in a study of terpene compounds in cheeses produced by cows grazing on different pastures showed that the terpene quantity is correlated to botanical composition of pasture and these compounds are more abun-

* Corresponding author. Tel.: +39 011 6708705; fax: +39 011 6708549.
E-mail address: giuseppe.zeppa@unito.it (Z. Giuseppe).

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dant in the highland richer in dicotyledons than in the lowlands richer in monocotyledons.

Monoterpenes and sesquiterpenes could be transferred from plant to milk in two ways: digestive and respiratory. In the first case the molecules pass to the plants in the rumen where a chemical transformation of some terpenes in other terpenes could be possible. All these molecules pass to the rumen into the blood then into the milk. Terpenes from herbs as well as terpenes produced in the rumen may be present [7]. In the second case compounds spread in the air are adsorbed by the lungs then transferred to the blood [8]. The transfer of these compounds to the milk can change its composition and particularly its microbiological and aroma characteristics [6,8–11]. Several studies were then conducted to define a relation between cheese and its production area with the study of animal feed and above all with the study of terpene compounds [4–6,8,9,11–14]. Results showed that milk from different production sites (lowland versus highland) and seasons (winter versus summer) can be distinguished [6,9,10] but there is no research devoted to differentiate the production site located in the same area. As the obtained products from highland pastures in the same valley are very different, differentiation using mono- and sesquiterpenes of these products could be possible. The aim of this work was to use mono- and sesquiterpene composition obtained by headspace solid-phase microextraction–gas chromatography–mass spectrometry (HS-SPME–GC–MS) to define a traceability of cheese and the identification of producer. For this purpose, an artificial neural networks (ANNs) with the terpene compounds as input neurons and the mountain farms as output neurons were used.

The ANNs were chosen because they are able to learn intelligently through an automatic process and define a model that can be used as an ‘identity card’ for the classification of unknown samples and the identification of the cheese-maker.

2. Experimental

2.1. Samples

The study was conducted with a ricotta cheese named ‘Saras del Fèn’ produced only during the summer in the alpine farms located in a small alpine valley of Piedmont (North West Italy).

The Saras del Fèn is produced using a mixture of cow and/or sheep and/or goat whey added to cow and/or sheep and/or goat milk (0–15%). The whey is heated gradually at 60–70 °C. At this temperature the whey is added to the mixture of milks and then heated to 80 °C. Once this temperature is reached, the whey is coagulated with citric acid or magnesium sulphate. When the curd has formed, the whey–curd mixture is heated to 90–95 °C, then the curd is finally removed, salted and placed in linen cloths. These bags are hung for 24–48 h forming a characteristic half-sphere shape. At the end of this time, the ricotta cheese is removed from the linen cloths, if necessary salted dry and ripened for at least 21 days at 8–12 °C in curing rooms.

This research was conducted during the summer 2003 and the products of nine mountain farms were analysed (Table 1).

During the mountain grazing, approximately every 7 days a sample of ricotta cheese was taken from each farm, put into vacuum-packed polyethylene bags and stored at −20 °C. As mountain grazing differs among producers, the first sample was collected 4 or 5 days after arrival in the highlands to allow the rumen emptying and as some cheese-makers produce this ricotta cheese sporadically, the sample number changes between farms.

2.2. HS-SPME analysis

The ricotta cheese samples were conditioned to ambient temperature before analysis then a sample of 20 g was taken out, placed in a beaker and homogenized. 2.5 g of this cheese was placed in a 10 mL pre-assembled clear glass vial (38 mm high and 22 mm in diameter) capped with a 20 mm PTFE/silicone septa (Supelco, Bellefonte, PA, USA). The vial was placed in a water bath at 53 °C and stirred for 10 min for equilibrium. After this time the SPME fibre was positioned in the headspace. The SPME fibre used for the study was a StableFlex 2 cm—50/30 μm divinylbenzene–carboxen–polydimethylsiloxane (DVB–CAR–PDMS) (Supelco). After exposure during 1 h, the fibre with the concentred analytes was retracted and transferred to the injector that was operated in the splitless mode at a temperature of 270 °C for 6 min. Different extraction temperature (30, 35, 38, 40 and 53 °C) and extraction time (20, 40, 45 and 60 min) were tested for an optimisation of method and the highest absorption of mono- and sesquiterpenes according to Kovačevič and Kač [15]. Three replicated analysis were performed for each sample.

2.3. GC–MS analysis

Compound identification was achieved with a Shimadzu GC-17A gas chromatograph (GC) coupled with a Shimadzu QP-5000 quadrupole mass spectrometer (Shimadzu Corporation, Kyoto, Japan).

The GC was equipped with a DB-WAX fused silica capillary column, 30 m × 0.25 mm i.d., 0.25 μm film thickness (J&W Scientific Inc., Folsom, CA, USA) and a split–splitless injector. The carrier gas was ultrahigh purity (99.999%) helium with a flow rate of 1.0 mL/min. The following column temperature programming sequence was used: an initial temperature of 35 °C for 5 min, increased to 173 °C at a rate 2 °C/min for an additional 1 min then increased to 210 °C at a rate of 15 °C/min and an additional 5 min. Mass spectra were recorded in the electron impact mode at an ionisation voltage of 70 eV in the 33–300 amu mass range. The ion source and the interface were maintained at 220 °C. The scan rate was 500 amu/s, start time 1.0 min. Compound identification was carried out with the mass spectra and retention times

Table 1
Characteristics of mountain farms examined and related identification codes

Farm name	Identification code	Altitude (m)	Pasture surface (ha)	Herd composition
Alpe Gianna	G	1680–2620	748	120 cows + 20 goats
Alpe Pis Uvert	C	1730–2400	1227	8 cows + 100 sheeps
Alpe Prà Inferiore	CT	1700–2400		36 cows
Alpe Bancet	B	1950–2780	514	16 cows + 100 goats
Alpe Vandalino	PO	1500–2100	210	10 cows + 10 goats
Partia d'Aumont	P	1700–2650	1470	400 sheeps + 75 goats
Partia d'Aumont	R	1700–2650	1470	60 cows + 25 sheeps + 5 goats
Alpe Palà	T	1570–2180	640	50 cows + 70 goats
Alpe Chiot d'la Sella	M	1200–2150	110	25 cows + 85 sheeps + 10 goats

of standard compounds, when available, or/and the NIST 12, NIST62 (National Institute of Standards and Technology, Gaithersburg MD, USA) and Adams [16] mass spectral data base and calculate the retention indexes.

Quantitative analysis of all monoterpenes and sesquiterpenes was performed integrating with a Class-5000 Data Station Ver. 2.0 (Shimadzu) the intensity of the base peak ion or characteristic peak ion in total ion current (TIC).

2.4. Chemometric processing

For each sample and each terpene, the intensity mean value of the base peak or characteristic ion in TIC peak of the three replications was calculated. Whenever the terpene was absent a "0" value was included and subsequently used in the statistical analysis. To highlight the differences between the farms, a variance analysis of each mono-and sesquiterpene detected was performed (Statistica for Windows Ver. 6, Tulsa, USA).

Artificial neural networks were generated with NeuroShell, Rel. 2 (Ward System Groups Inc., Frederick, MD, USA). The network architecture used was a three-layer, fully interconnected, feed-forward type. Decision on the number of hidden layers to use is complex as it depends on the specific problem being solved using ANN. Different hidden layer numbers and nodes in the hidden slabs were chosen but these variations did not influence the network learning level. Afterwards the default three layers network with 21 nodes in the input, 22 in the hidden and 9 in the output layer were used for the monoterpenes and a network with 16 nodes in the input, 22 in the hidden and 9 in the output layer was used for the sesquiterpenes. Also different combinations of learning rate and momentum were tested (not reported data) and the best prediction results were obtained with a learning rate of 0.1 and a momentum of 0.1. For the input slab, the linear activation function was used while the logistic functions were used for hidden and output layer slabs. Inputs of the data sets were normalised in the $0 \div 1$ range before use in training and testing of the ANN. To avoid network overtraining, NET-PERFECT was used. This is an implemented procedure of NeuroShell 2 that creates an entirely separate set of data, called test-set, and uses it to evaluate how well the network is predicting. NET-PERFECT was used to compute the optimum point for saving the network when it is able to generalise new data well. Testing data were fed to test trained ANN after training 200 epochs. In particular, the network learning was carried out with a limit of 200,000 events after the test set's minimum mean value of re-classification error was reached. For ANNs all the samples were used. These samples were randomly subdivided into a training set (70%) and a validation set (30%). In the training set, therefore, there are approximately 92 samples and in the validation set 40 samples. The distribution percentage of the samples between the two data sets was chosen empirically and represents a compromise between the need to have the maximum number of samples in the training set while at the same time representing all product categories in the validation set. The ANN construction process, from the two data set extractions through learning, was repeated five times.

3. Results and discussion

3.1. Optimization of SPME method

SPME was largely used for analysis of volatile compounds of cheese [17–25] but there are no reported indications for terpene determination. So the fiber used was chosen on the suggests of Supelco for the volatile and semivolatile flavour compounds with C3–C20. As reported by Kovačevič and Kač [15] with higher extraction temperatures and longer extraction time the fibre concentrations of less volatile compounds increase while those of more volatile compounds decrease. For myrcene and α-humulene these Authors showed that with a polydimethylsiloxane fibre and the hop aroma the most suitable conditions are 30 min at 70 °C but even after 4 h the system does not reach equilibrium thus extraction time can be longer. The choice of 30 min is a compromise for routine work. Similar results were highlighted by Pinho et al. [24] where the extraction efficiency of a Carboxen-polydimethylsiloxane fibre used for the cheese aroma is maxima with an adsorption time of 60 min at 20 °C. According to these results in our work some trials were performed to define the extraction conditions. The α-pinene and the limonene were used as reference monoterpenes and the α-copaene and the α-caryophyllene as reference sesquiterpenes. A sample of ricotta cheese was analysed at different pairs of temperature and time (Table 2). The intensity of the base peak ion in total ion current for these compounds increase with long

Table 2
Intensity of the base peak ion in total ion current for two terpenes and two sesquiterpenes at different pairs of temperature and time extraction

	DVB/carb/PDMS				
	20 min at 35 °C	40 min at 30 °C	45 min at 38 °C	1 h at 40 °C	1 h at 53 °C
Monoterpenes					
α-Pinene	6.943.926	11.157.454	16.793.199	12.895.044	20.717.130
Limonene	174.941	191.993	315.894	433.250	1.004.855
Sesquiterpenes					
α-Copaene	0	20.071	36.758	54.877	218.432
α-Caryophyllene	0	0	52.886	93.258	287.369

Table 3
Monoterpenes and sesquiterpenes detected in ricotta samples of each mountain farm and result of variance analysis performed for each detected terpene between the nine farms

Number	Name	Rt	Rl	Mountain farms									
				B	G	P	PO	R	CT	M	C	T	Statistical significance
Monoterpenes													
1	Monoterpene (ni 1)	6.4	981	*	*	*				*	*	*	**
2	α-Pinene	7	1001	*	*	*	*	*	*	*	*	*	**
3	Monoterpene (ni 2)	7.3	1007	*	*	*		*		*	*	*	**
4	Monoterpene (ni 3)	8.4	1029			*						*	**
5	Camphene	8.8	1037	*	*	*	*	*	*	*	*	*	**
6	β-Pinene	10.5	1071	*	*	*	*	*	*	*	*	*	**
7	Sabinene	11.3	1087	*	*	*	*	*		*	*	*	**
8	δ-3-Carene	12.7	1112	*	*	*	*	*			*	*	**
9	Monoterpene (ni 4)	13.8	1129	*		*	*	*			*	*	**
10	β-Myrcene	14.1	1134	*		*		*		*	*	*	**
11	Monoterpene (ni 5)	14.9	1146	*	*	*		*	*	*	*	*	**
12	Limonene	16	1164	*	*	*	*	*	*	*	*	*	**
13	β-Phellandrene	16.5	1172	*	*	*		*		*	*	*	**
14	γ-Terpinene	19.2	1213	*	*	*	*	*		*	*	*	**
15	*p*-Cymene	20.8	1237	*	*	*	*	*	*	*	*	*	**
16	Monoterpene (ni 6)	21.8	1252	*	*	*	*	*	*	*	*	*	**
17	Monoterpene (ni 7)	28	1343			*							**
18	Linalool	40	1527	*	*	*	*	*	*		*	*	**
19	Bornyl acetate	41.3	1547	*								*	**
20	Verbenone	48.1	1657	*		*	*			*		*	**
21	α-Terpineol	48.7	1667	*	*	*	*				*	*	**
22	Myrtenol	53.8	1757	*	*	*		*			*	*	**
Sesquiterpenes													
23	α-Copaene	35.3	1454	*	*	*		*		*	*	*	**
24	β-Maaliene	37	1480			*							**
25	Isocomene	37.8	1492			*							**
26	Sesquiterpene (ni 1)	38.4	1501			*							**
27	Selinan	41.6	1552	*	*	*					*	*	**
28	9-epi-Caryophyllene	41.7	1554	*	*	*	*	*	*	*	*	*	**
29	Sesquiterpene (ni 2)	42.2	1562	*	*	*				*	*	*	**
30	Sesquiterpene (ni 3)	44.2	1594	*		*					*	*	**
31	α-Caryophyllene	46	1623	*	*	*	*	*		*	*	*	**
32	epi-Cedrane	46.6	1633	*	*	*		*	*	*	*	*	**
33	Sesquiterpene (ni 4)	47.5	1648	*	*	*		*			*		**
34	Sesquiterpene (ni 5)	47.9	1654	*		*					*	*	**
35	Isocaryophyllene	49	1672	*	*	*			*			*	**
36	Sesquiterpene (ni 6)	49.3	1677	*	*	*							**
37	Sesquiterpene (ni 7)	51.5	1715	*	*	*				*	*	*	**
38	Valencene	52.4	1831	*	*	*					*	*	**

For mountain farm code see Table 1. ni, Not identified; Rt, retention time; RI, retention index; *, presence of the compound.
** $P < 0.01$Statistical significance.

time and high temperature. These results are opposite to those of Kovačevič and Kač [15] but the fibres used are different. Similar fibre and condition are also used by Bellesia et al. [18] (80 °C and 30–35 min) and Costa et al. [25] (60 °C and 50 min).

3.2. Repeatability

The method repeatability was defined by analyzing seven samples of ricotta cheese using 2.5 g of sample, an extraction time of 1 h and a temperature of 53 °C. The relative standard deviation ranged between 1% for sesquiterpene (ni 6) and 18% for sabinene.

3.3. Cheese traceability

In all 22 mono- and 16 sesquiterpenes were detected (Table 3). Depending on the mountain farm and grazing period the total number of mono- and sesquiterpenes varied from 27 to 37.

The most widespread monoterpenes are α-pinene, camphene, β-pinene, δ-3-carene, limonene already reported

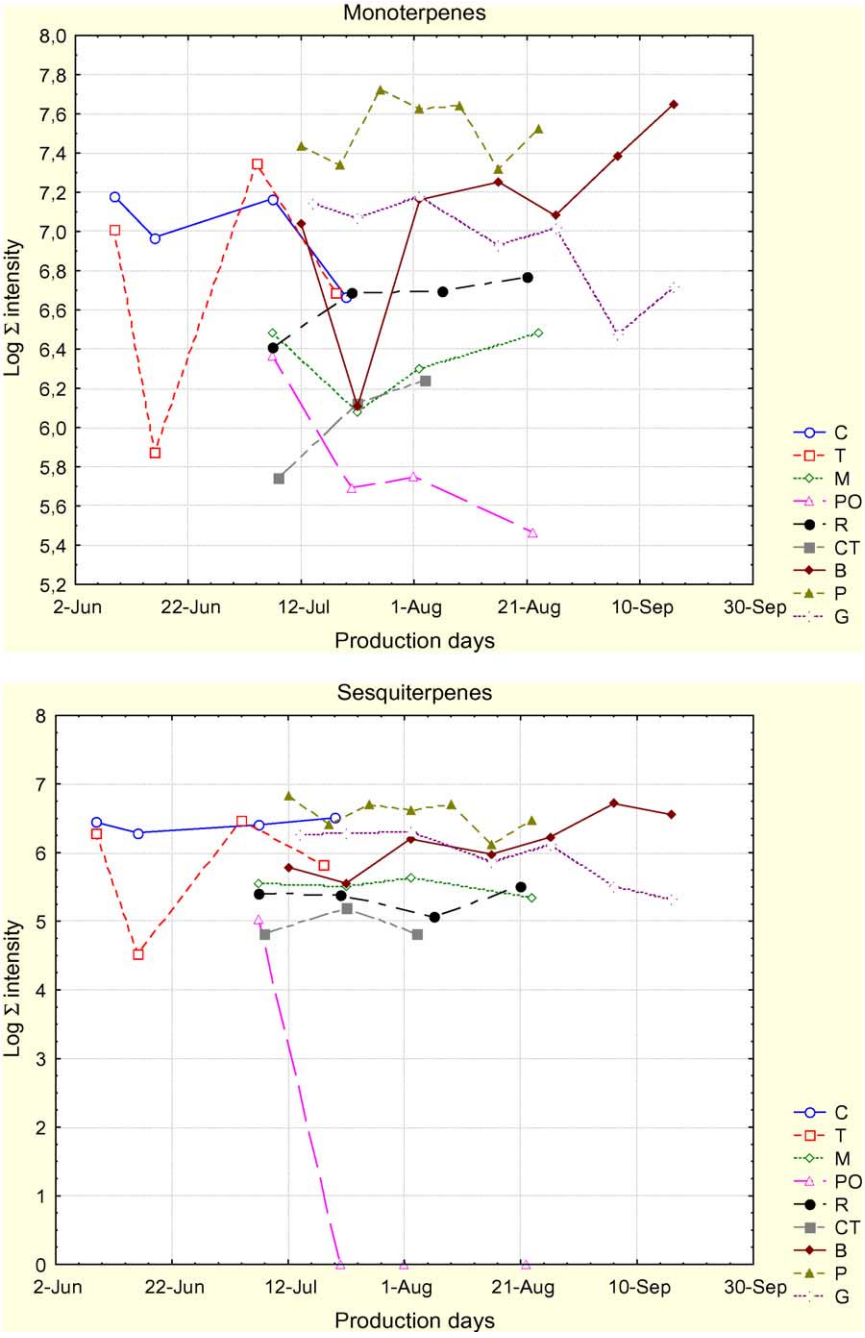

Fig. 1. Mono- and sesquiterpene evolution for each farm during the mountain grazing. For each sample, the reported values are the sum of intensity of base peaks evaluated in TIC of all mono- and sesquiterpenes detected.

in some alpine grassland herbs (*Heracleum sphondylium*, *Ligusticum mutellina*, *Aposeris foetida*, *Aster bellidiastrum*, *Leucanthemum vulgare*, *Geranium sylvaticum*, *Mentha longifolia*, *Meum athamanticum*, *Pimpinella saxifraga*, *Achillea millefolium*) [4,13]. For the sesquiterpenes the most important are α-copaene, selinan, 9-epi-caryophyllene, α-caryophyllene, isocaryophyllene which were already reported in alpine grassland herbs (*Heracleum sphondylium*, *Ligusticum mutellina*, *Leucanthemum vulgare*, *Prunella vulgaris*, *Pimpinella saxifraga*) [4,13].

All these compounds showed a wide concentration variability due above all to the different composition of herds, but in each farm the herd changed pasture during the mountain grazing, according to the vegetative phase of herbs thus the differences for type and concentration of mono- and sesquiterpene compounds are given also to the different *facies* in the pasture and the vegetative phase of herbs eaten (Fig. 1).

The P farm is characterised by the greatest quantity of mono- and sesquiterpenes. This could be due to the presence of only sheep and goats. These animals have a wide range of pastured herbs, more varied than cows, and are very mobile in the pasture to search young herbs that are very rich in terpene compounds [4,26]. On the contrary the PO farm is characterised by the lowest quantity of terpene compounds. In particular sesquiterpenes are absent in the ricotta samples after July 30th. Then the cow herd of PO farm pastured in this period in *facies* with low production of monoterpenes and no sesquiterpenes. The B farm is of particular interest where mono- and sesquiterpene quantities increased during the mountain grazing. This is due to the quantity difference between cow and goat milks produced on this farm. With the progress of mountain grazing cow milk decreased and the goat milk quantity increased. Goats have a more varied diet and rich in herbs compared to that of cows and above all with large amounts of dicotyledons rich in terpene compounds. The T farm is also interesting with a high reduction of mono- and sesquiterpene concentrations in the second sample. This is correlated to the herd's move to pastureland. The first sample corresponds to the herd's arrival in a pasture with a large amount of dicotyledons. Then the herd transferred near to the farm where the monocotyledons (second sample) were more abundant. At the end of June the herd transferred to the high pastures very rich in dicotyledons where they remained until the return to valley.

For the other farms it is not possible to explain the mono- and the sesquiterpene evolution because there is no information about the vegetable composition of their pasture land.

Despite this concentration variability during pasturing, the variance analysis performed for every terpene detected highlights a statistical difference between the nine mountain farm ricotta cheeses (Table 3). A difference test (Duncan test) was not performed because the aim of this work was the construction of a predictive model and not simply a farm differentiation. This difference can be obtained with the ANN results in which the mean reclassification value is 99% for the monoterpenes and 93% for the sesquiterpenes (Table 4). The most important compounds for the

Table 4
Mean reclassification value (%) for each mountain farm calculated with five cycles of learning using only the sesquiterpene or the monoterpene compounds

Mountain farm	Sesquiterpenes	Monoterpenes
B	97	97
C	100	100
CT	95	100
G	97	100
M	100	100
P	99	100
PO	83	98
R	100	96
T	66	100

Table 5
Mean values of relative importance of artificial neural network input (contribution factors) calculated for each compound after five learning cycles

Name	Contribution factor
Monoterpenes	
Monoterpene (ni 1)	0.675134
Bornyl acetate	0.597056
Camphene	0.564604
Verbenone	0.531416
Myrtenol	0.488568
α-Pinene	0.444406
p-Cymene	0.43512
Monoterpene (ni 2)	0.430402
α-Terpineol	0.419758
δ-3-Carene	0.417496
Monoterpene (ni 3)	0.394086
β-Myrcene	0.385868
Limonene	0.365614
Linalool	0.350134
Sabinene	0.347238
β-Pinene	0.331266
γ-Terpinene	0.32217
Monoterpene (ni 7)	0.320858
Monoterpene (ni 5)	0.308526
β-Phellandrene	0.308196
Monoterpene (ni 4)	0.288458
Monoterpene (ni 6)	0.273624
Sesquiterpenes	
Sesquiterpene (ni 4)	0.755126
Sesquiterpene (ni 6)	0.75118
α-Caryophyllene	0.724208
Selinan	0.68058
9-epi-Caryophyllene	0.673554
Sesquiterpene (ni 7)	0.664054
epi-Cedrane	0.628718
Valencene	0.566668
Isocaryophyllene	0.563184
Sesquiterpene (ni 5)	0.516798
Sesquiterpene (ni 3)	0.491316
Sesquiterpene (ni 2)	0.461632
α-Copaene	0.454598
Sesquiterpene (ni 1)	0.386988
β-Maaliene	0.341422
α-Isocomene	0.33998

ANNs are monoterpene (ni 1), bornyl acetate, camphene and verbenone for the monoterpenes and sesquiterpene (ni 4), sesquiterpene (ni 6), α-caryophyllene, selinan, 9-epi-caryophyllene, sesquiterpene (ni 7), epi-cedrane, valencene, isocaryophyllene and sesquiterpene (ni 5) for the sesquiterpenes (Table 5).

The average learning is very high for monoterpenes (99%) where only some "R" samples are classified as "PO" and some "B" samples as "G".

On the contrary more problems in ANN self-configuration with re-classification errors are reported using the sesquiterpenes as input variables. The highest problems are highlighted for the "T" samples, classified as "CT" or "PO" and for the "PO" samples classified as "CT".

Probably this is due to the *facies* of these highlands with a similar production of sesquiterpenes, but there are no data for the plants present in these mountain farms; thus, it is not possible to understand the reported errors in ANNs.

4. Conclusion

Monoterpenes and sesquiterpenes can be used not only to distinguish between summer pasture cheeses and the ones produced during other periods as reported by more Authors but can be used also as markers for each mountain pasture. ANNs could be very important for this approach since they constitute an efficient tool for characterising and discriminating food products. Moreover the ANNs could be used for the classification of samples not taken into account during learning for the control of commercialised products. But for a good interpretation of differences between mountain farms it is necessary to know exactly the botanical composition of the highlands and above all the mono- and sesquiterpene composition of each *facies* plant and their evolution according to phenological phase.

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