

## Compositional, microbiological, biochemical, volatile profile and sensory characterization of four Italian semi-hard goats' cheeses

Raffaella Di Cagno<sup>1</sup>, R Evan Miracle<sup>2</sup>, Maria De Angelis<sup>1</sup>, Fabio Minervini<sup>1</sup>, Carlo G Rizzello<sup>1</sup>, Mary Anne Drake<sup>2</sup>, Patrick F Fox<sup>3</sup> and Marco Gobbetti<sup>1\*</sup>

<sup>1</sup> Dipartimento di Protezione delle Piante e Microbiologia Applicata, Università degli Studi di Bari, Italy

<sup>2</sup> Southeast Dairy Foods Research Center, Department of Food Science, North Carolina State University, Raleigh, NC USA

<sup>3</sup> Department of Food and Nutritional Sciences, University College Cork, Cork, Ireland

Received 22 December 2006 and accepted for publication 3 July 2007

Four semi-hard Italian goats' milk cheeses, Flor di Capra (FC), Caprino di Cavalese (CC), Caprino di Valsassina (CV) and Capritilla (C), were compared for compositional, microbiological, biochemical, volatile profile and sensory characteristics. Mean values for the gross composition in part differed between cheeses. At the end of ripening, cheeses contained 7.98–8.51 log<sub>10</sub> cfu/g of non-starter lactic acid bacteria. *Lactobacillus paracasei*, *Lb. casei* and *Lb. plantarum* were dominant in almost all cheeses. As shown by the Principal Component Analysis of RP-FPLC data for the pH 4.6-soluble fractions and by the determination of free amino acids, secondary proteolysis of CC and CV mainly differed from the other two cheeses. A total of 72 volatile components were identified by steam distillation-extraction followed by gas chromatography-mass spectrometry. Free fatty acids and esters qualitatively and quantitatively differentiated the profile of CV and CC, respectively. The lowest concentrations of volatile components characterized FC. Descriptive sensory analysis using 17 flavour attributes was carried out by a trained panel. Different flavour attributes distinguished the four goats' cheeses and relationships were found with volatile components, biochemical characteristics and technology.

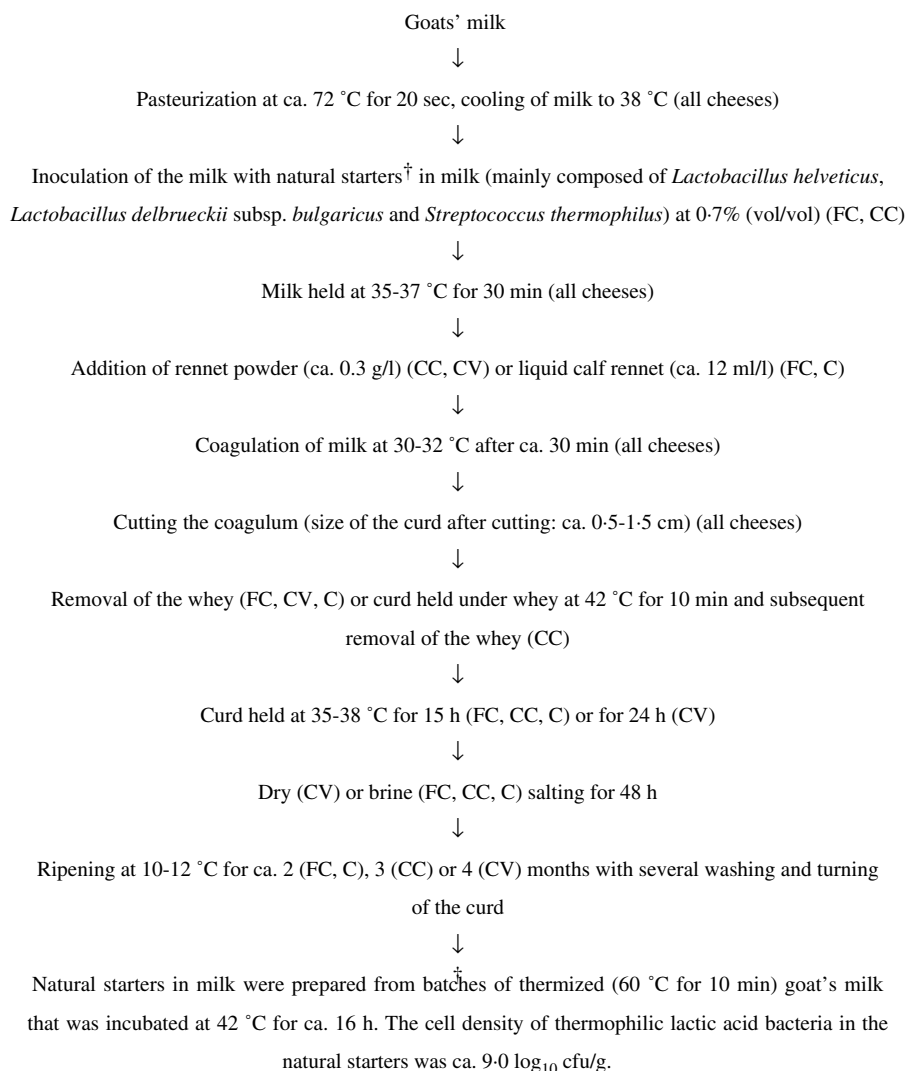
**Keywords:** Goats' cheeses, non-starter lactic acid bacteria, proteolysis, volatile components, sensory analysis.

After France, Greece and Spain, Italy is the fourth country in the EU for goats' milk production, ca. 115 000 t per year (FAOSTAT, 2004). Goat-farming is located mainly in the Alps, Centre-Southern and the major islands (Sardinia and Sicily) of Italy. As a consequence of these locations in heterogeneous zones, many different goat breeds are present. Except for minor local production of goats' milk yogurt or pasteurized goats' milk, all the milk is transformed into cheese, alone or mixed with cows' and/or ewes' milks. Semi-hard varieties, made from predominantly rennet-coagulated curd, are larger than soft cheeses, have a cylindrical shape and a dry rind, and consumers' demand is increasing rapidly owing to typical texture and flavour.

Contrary to some of the most popular French and Spanish goats' cheeses which have a Protected

Denomination of Origin (PDO) and were characterized in a number of studies (Medina & Nuñez, 2004), none of the Italian goats' cheese has PDO status and studies are few. The few reports have considered the microbial characterization of fresh (Casalta et al. 2001) or semi-hard goats' cheese (Suzzi et al. 2000) and the use of various levels of rennet and of mesophilic starters to increase the cheese yield (Caponio et al. 2001). Studies on the compositional, microbiological, biochemical, volatile profile and sensory characteristics of the most important Italian semi-hard goats' cheeses may have the following objectives: (i) to differentiate cheeses; (ii) to establish the effect of selected technological parameters on specific differences in the microbiota and related biochemical activities; and, in general, (iii) to identify the most appropriate characters suitable for obtaining a 'denomination of origin'. This study characterizes four major typical Italian semi-hard goats' cheeses, Flor di Capra, Caprino di Valsassina, Caprino di Cavalese and Capritilla.

\*For correspondence; e-mail: gobbetti@agr.uniba.it



**Fig. 1.** Protocols for the manufacture of the four Italian goats' cheeses. FC, Flor di Capra; CC, Caprino di Cavalese; CV, Caprino di Valsassina; C, Capritilla.

## Materials and methods

### Cheese samples

Flor di Capra (FC; from milk of the Sarda breed, Sardinia region); Caprino di Cavalese (CC; Saanen breed, Trentino Alto Adige region); Caprino di Valsassina (CV; Orobica breed, Trentino Alto Adige region); and Capritilla (C; Alpina breed, Piemonte region) were supplied in triplicate (different batches from the same factory) (Fig. 1). Cheeses were manufactured during spring and milk was from the early lactation. Cheese manufacture was according to specific guidelines for each variety (<http://www.formaggio.it>). The gross compositions of the goats' milks were: protein, 2.9 (Saanen)–4.2% (Sarda); fat, 2.9 (Orobica)–4.0% (Sarda); and lactose, 4.3–4.5%. The pH was 6.6–6.7. All cheeses had cylindrical shape with a diameter of 19–21 cm, height of 6–9 cm and weight 2–3 kg.

All the analyses were carried out in duplicate for each batch of cheese.

### Compositional and microbiological analyses

Moisture, NaCl, pH and the pH 4.6-soluble nitrogen (expressed as a % of total nitrogen in the cheese) were determined as described by Di Cagno et al. (2003). Protein and fat were determined as described by Gobbetti et al. (1999).

Thermophilic streptococci were enumerated on M17 (Oxoid Ltd, Basingstoke, Hampshire, England) at 42 °C for 72 h. Mesophilic and thermophilic lactic acid bacteria (LAB) were enumerated on MRS agar (Oxoid Ltd) after incubation at 30 or 42 °C for 72 h. At least 15 colonies of presumptive mesophilic LAB were isolated from the plate of the highest dilution of each type of cheese

**Table 1.** Gross composition of the four Italian goats' cheeses at the end of ripeningValues are means  $\pm$  SD for three batches of each type of cheese, analysed in duplicate

	Flor di Capra	Caprino di Cavalese	Caprino di Valsassina	Capritilla
Moisture (% wt/wt)	34.0 $\pm$ 0.6 <sup>a</sup>	31.3 $\pm$ 0.9 <sup>b</sup>	28.2 $\pm$ 0.5 <sup>c</sup>	33.8 $\pm$ 0.8 <sup>a</sup>
Fat (% wt/wt)	30.3 $\pm$ 0.2 <sup>b</sup>	33.1 $\pm$ 0.5 <sup>a</sup>	32.7 $\pm$ 0.4 <sup>a</sup>	32.8 $\pm$ 0.7 <sup>a</sup>
NaCl (% wt/wt)	0.6 $\pm$ 0.01 <sup>b</sup>	1.0 $\pm$ 0.05 <sup>a</sup>	1.1 $\pm$ 0.03 <sup>a</sup>	0.8 $\pm$ 0.01 <sup>b</sup>
Protein (% wt/wt)	23.0 $\pm$ 0.5 <sup>c</sup>	26.0 $\pm$ 0.6 <sup>b</sup>	27.5 $\pm$ 0.3 <sup>a</sup>	23.5 $\pm$ 0.7 <sup>c</sup>
pH	5.00 $\pm$ 0.2 <sup>c</sup>	5.30 $\pm$ 0.1 <sup>a</sup>	5.21 $\pm$ 0.3 <sup>b</sup>	5.19 $\pm$ 0.1 <sup>b</sup>
pH 4.6-soluble N (% of total N)	20.1 $\pm$ 1.1 <sup>c</sup>	24.2 $\pm$ 0.8 <sup>a</sup>	24.5 $\pm$ 0.8 <sup>a</sup>	22.2 $\pm$ 1.0 <sup>b</sup>

<sup>a-c</sup> Means within a row with different superscript letters are significantly different ( $P < 0.05$ ).

and subjected to genotypic identification. Extraction of genomic DNA and primers, LacbF/LacbR and LpCoF/LpCoR (Invitrogen Life Technologies, Milan, Italy), used to amplify 16S rRNA gene fragment were as described by Coda et al. (2006). The expected amplicons of about 1400 and 1000 bp were eluted from the gel and purified by the GFX<sup>TM</sup> PCR DNA and Gel Band Purification Kit (Amersham Pharmacia Biotech AB, Uppsala, Sweden). Taxonomic strain identification was carried out by using the Basic BLAST database. Strains showing homology of at least 97% were considered to belong to the same species.

#### Assessment of proteolysis

The pH 4.6-soluble fractions of the cheeses were prepared as described by Gobbetti et al. (1999) and analysed by Reverse-Phase Fast Protein Liquid Chromatography (RP-FPLC) with a Resource RPC column, using an ÄKTA FPLC equipment with a UV detector operating at 214 nm (Amersham). Peptide profiles were analysed by multivariate statistical techniques. The data for factor reduction analysis were obtained by visually recognizing the peaks and taking peak heights as variables (Pripp et al. 1999). Total and individual free amino acids (FAA) in the pH 4.6-soluble fraction were analysed using a Biochrom 30 series Amino Acid Analyzer (Biochrom Ltd., Cambridge Science Park, England) as described by Coda et al. (2006).

#### Determination of volatile components

Simultaneous steam distillation-extraction (SDE) was conducted to extract volatile compounds from cheese followed by gas chromatography mass spectrometry (GC/MS) for compound identification. Prior to analysis, the rind was trimmed and a wedge cross-section of cheese was taken. Samples were shredded and stored at  $-20^{\circ}\text{C}$  until extraction. Ten grams of frozen, grated cheese was mixed with 40 ml of deodorized water and boiling chips in a 100 ml

steam distillation flask. SDE was carried out for ca. 2 h into dichloromethane containing 10 ppm methylnonadecanoate as internal standard (Chrompack UK Ltd, Millharbour, London). An Agilent 6890 with 5973N mass selective detector, equipped with a fused silica capillary column (DB-5MS 30 m length  $\times$  0.25 mm i.d.  $\times$  0.25  $\mu\text{m}$   $d_f$ , J&W Scientific), was used for GC-MS analysis (Karagul-Yuceer et al. 2001). Based on MS results, relative concentrations of the compounds were calculated. The area ratio (area of internal standard/area of compound) was multiplied by the concentration of the internal standard to determine the relative abundance of the compounds. For positive identification, retention indices and mass spectra were compared with those of authentic standard compounds. For the calculation of retention indices, an n-alkane series was used.

#### Sensory analysis

For sensory analysis, the rind was trimmed from each cheese and cross-sectional wedges were taken. Cheeses were then sliced into 2  $\times$  2 cm cubes such that each panellist received at least 3 cubes that represented the entire cross-section of the wedge at each tasting session. Samples were evaluated by a trained cheese sensory panel ( $n=8$ ) using a previously published lexicon for cheese flavour (Drake et al. 2001) with specific descriptors for goats' cheeses added (Carunchia-Whetstine et al. 2003). Flavour and taste intensities were scaled using a 15-point universal intensity scale.

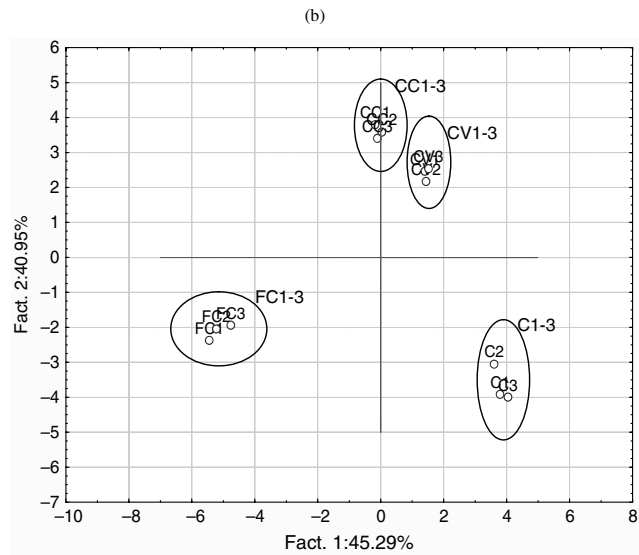
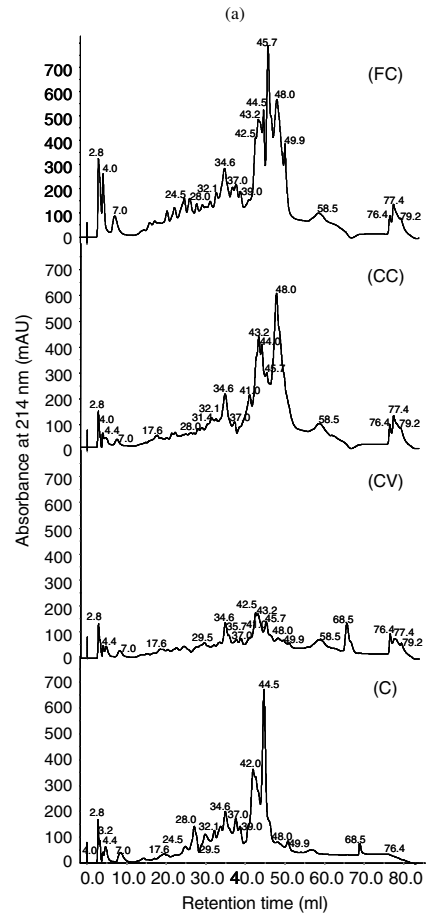
#### Results and discussion

The lowest and highest levels of moisture were found for CV and FC, which mainly agreed with the time of ripening (Table 1). The protein content varied by 4.5% between the four cheese types. Only FC had significantly lower fat than the other cheeses (Table 1). These ranges agreed with

those of other semi-hard goats' cheeses (Medina & Nuñez, 2004) and reflected the chemical composition of the raw goats' milk. The pH values varied by 0.3 between the four cheeses, indicating that this is not influenced by the thermophilic starters used. The level of NaCl ranged from 0.6–1.1% (wt/wt); it was highest for the dry salted CV. Overall, for this and the further analyses the same cheese type from different batches did not differ significantly ( $P>0.05$ ) showing the reproducibility of the protocols for the manufacture.

Numbers of thermophilic lactobacilli and streptococci in FC and CC cheeses that used thermophilic natural starters varied from  $5.10 \pm 0.35$ – $4.38 \pm 0.14$  and  $4.54 \pm 0.20$ – $4.92 \pm 0.36$   $\log_{10}$  cfu/g, respectively. Thermophilic LAB in the other two cheeses were less than  $3.0 \log_{10}$  cfu/g. Numbers of presumptive mesophilic LAB were similar between cheeses and varied from  $7.98 \pm 0.22$  (FC) to  $8.51 \pm 0.18$   $\log_{10}$  cfu/g (CV). The microbial composition was also similar and the following species were identified: FC, *Lb. paracasei* (12 isolates) and *Lb. casei* (3); CC, *Lb. paracasei* (12), *Lb. casei* (1), *Lb. plantarum* (1), and *Pediococcus pentosaceus* (1); CV, *Lb. paracasei* (6), *Lb. plantarum* (5), *Lb. rhamnosus* (3), and *Lb. casei* (1); and C, *Leuconostoc pseudomesenteroides* (6), *Lb. plantarum* (5), *Lb. paracasei* (3), and *Lb. casei* (1). *Lb. paracasei*, *Lb. plantarum* and *Lb. rhamnosus* were also found as the dominant species in several Spanish goat' cheeses (Mas et al. 2002; Sánchez et al. 2005). *Pediococcus* and *Leuconostoc* sp. were identified in Ibores (Mas et al. 2002) and other Spanish goats' cheeses (Fontecha et al. 1990).

The level of pH 4.6-soluble nitrogen (% of total nitrogen) varied by 4.4% between the four cheese types (Table 1). These values agreed with those found for the Italian Bastelicaccia and Spanish Ibores goats' cheeses (Casalta et al. 2001; Mas et al. 2002) and were markedly higher than those found for the Spanish and French goats' cheeses Babia-Laciana, Armada and Sainte Maure (Fresno et al. 1997; Le Quééré et al. 1998; Franco et al. 2001). The pH 4.6-soluble fractions were analysed by RP-FPLC (Fig. 2a) and subjected to Principal Component Analysis (Fig. 2b). CC and CV grouped together while FC and C were located in separated zones of the plane. The concentration of FAA was highest for CV ( $17.94 \pm 2.2$  mg/g) and CC ( $15.76 \pm 3.5$  mg/g), intermediate for FC ( $10.39 \pm 2.7$  mg/g) and lowest for C ( $5.39 \pm 1.2$  mg/g). These concentrations were higher than those found for the Spanish and French varieties Babia-Laciana (ca. 4 mg/g; Franco et al. 2001), Armada (ca. 3.6 mg/g; Fresno et al. 1997) and Bouton de Culotte (ca. 2.6 mg/g; Salles et al. 2000). Ser, Glu, Val, Ile, Leu, Tyr, Phe, Lys and Pro were found at the highest concentrations in the four Italian cheeses. This profile did not agree with that of Babia-Laciana cheese, mainly characterized by  $\gamma$ -amino butyric acid, Val and Leu (Franco et al. 2001). Only the concentrations of 5 FAA (Glu, Cys, Tyr, His and Trp) statistically ( $P<0.05$ ) differentiated the profiles of CV and CC. Since these cheeses showed the



**Fig. 2.** Reverse-Phase Fast Protein Liquid Chromatography (RP-FPLC) of the pH 4.6-soluble fractions of the four Italian goats' cheeses (a) and related Score plot of first and second principal components after Principal Component Analysis (b). FC, Flor di Capra; CC, Caprino di Cavalese; CV, Caprino di Valsassina; C, Capritilla. Numbers 1–3 refer to different batches of cheese.

highest level of pH 4.6-soluble nitrogen (24.2–24.5%) and the less abundant peptide profile, especially CV (Fig. 2a), a higher degradation of peptides to FAA during ripening was suspected. CV and CC were manufactured by rennet powder, subjected to curd cooking (CC) or to curd held at 35–38 °C for the longest time (CV), ripened for 3 or 4 months (Fig. 1) and contained the large spectrum of NSLAB species.

Compared with headspace techniques, SDE showed the ability to recover also higher molecular weight volatiles (e.g., fatty acids larger than decanoic acid and lactones) (Larrayoz et al. 2001). Solid Phase Micro-Extraction was used with some success for cheese volatile analysis but it lacks the ability to extract the above high molecular weight volatiles (Coda et al. 2006). Dynamic headspace (Purge and Trap) noted fewer total recovered volatiles in Manchego cheese when compared with SDE (Gomez-Ruiz et al. 2002). Seventy-two volatile components were identified in the four Italian goats' cheeses by SDE (Table 2). Acids formed the most important group by concentration (21 components) (Table 2). The typical goat cheese aroma is attributed to short- and medium-chain linear, and branched-chain free fatty acids (FFA) (Medina & Nuñez, 2004). The concentrations of butanoic, nonanoic, decanoic, dodecanoic, tridecanoic, tetradecanoic, pentadecanoic, n-hexadecanoic and 9-octadecanoic acids differentiated CV from the other cheeses. Almost the same components, together with 3-methyl-butanoic, hexanoic, heptanoic and octanoic acids, statistically differentiated CC. Overall, a high variability characterized the qualitative profile of FFAs of Spanish goats' cheeses (Guillen et al. 2004b; Poveda & Cabezas, 2006). When rennet paste or other exogenous lipases are not used, the liberation of hexanoic to dodecanoic FFA is attributed to milk endogenous lipoprotein lipase (LPL) (Medina & Nuñez, 2004). LPL has a specificity towards fatty acids located at the positions sn-1, 3 of the triglyceride, and medium- and, especially short-chain fatty acids are predominantly esterified at the sn-3 position (Juárez et al. 1986). Besides, shorter chain triglycerides are more readily hydrolyzed by LPL than long-chain triglycerides because of higher solubility and mobility at the emulsion-water interface (Deckelbaum et al. 1990). Although the pasteurization included in the manufacture of all four goats' cheeses might reduce the activity of the LPL, its effect during milk storage and, especially, during prolonged ripening should be suspected (Morgan & Gaborit, 2001). The concentration of acids in CV was markedly higher than in the other Italian cheeses. Although technological traits such as curd held at 35–38 °C for 24 h and the longest time (4 months) of ripening had an influence, differences in the LPL activity due to the type of goats' milk breed might not be excluded. As shown for the goats' Palmero cheeses (Guillen et al. 2004a, b), a low number of alcohols were also identified in the four Italian cheeses (Table 2). 11-Dodecenol, 2-methyl-2-buten-1-ol and 9-octadecen-1-ol statistically differentiated the profile of CV. The same was found for

2-methyl-2-buten-1-ol, 4-methyl-phenol and phenylethyl alcohol in CC. A few aldehydes were identified (Table 2) which agreed with those reported in Palmero smoked goats' cheese (Guillen et al. 2004a). Esters, comprising 17 components, were the second most numerous chemical class (Table 2). Ethyl-hexanoate, 2-butyl-octanoate, isobutyl-caproate and 2-tridecyl-valerate statistically differentiated CV. Twelve of the 17 esters statistically differentiated CC from the other cheeses. Esters, especially those containing few carbon atoms, contribute in a synergistic way to the fruity aroma of cheese (Preininger & Grosch, 1994). Among ketones, only 2-heptanone and acetophenone statistically distinguished CV and CC from the other cheeses, respectively (Table 2). All the 5 aliphatic linear ketones identified in the Italian goats' cheeses were also found in Palmero goats' cheese (Guillen et al. 2004b).  $\gamma$ -Nonalactone,  $\delta$ -decenolactone and  $\delta$ -undecalactone were the most abundant lactones in all four goats' cheeses (Table 2). These compounds, probably resulting from oxidation of FFA and subsequent cyclation of the formed hydroxyacids, are cheese flavour components giving pleasant, buttery and fruity sensory attributes (Guillen et al. 2004a). Quantitatively, CV also contained alcohols, esters, ketones and lactones at the highest concentrations. The occurring enzymatic and chemical reactions seemed to be mainly related to the indigenous microbiota and, especially, to the time of ripening. This was confirmed by FC cheese (1 month of ripening) that instead of the use of primary starters had the poorest volatile profile.

Salty taste was scored between 3.0 and 4.3 (Table 3) and mirrored the differences found for the concentration of NaCl. FC was the only cheese with 7 flavour attributes not detectable and with appreciable intensities of whey and diacetyl, which are typical of fresh young cheeses (Drake et al. 2001). Besides, FC received the highest score for milk/fat and waxy/animal. This cheese was characterized by the lowest concentration of FFA and other volatile components (Table 2), shortest duration of ripening (Fig. 1) and less heterogeneous NSLAB composition. CC was characterized mainly by the attributes cowy/phenolic and brothy (aromatics associated with boiled meat). This cheese contained the highest amount of sulphur and aromatic compounds, including indole (Table 2) and was the only one subjected to curd cooking (Fig. 1). The attribute FFA was indubitably the characterizing flavour for the CV cheese. Indeed, its concentration of medium chain FFA was markedly higher than the other cheeses (Table 2). Due to the highest concentration of esters, especially with few carbon atoms, this cheese had also the highest score for fruity flavour. The flavour attribute amine/proteolytic received the highest score for C cheese. The only amine (4-methoxy benzenamine) identified among the volatile components statistically differentiated it from the other cheeses (Table 2).

First this study gave a complementary characterization of four major Italian semi-hard goats' cheeses. This

**Table 2.** Volatile components [area ratio (area of internal standard (ISTD)/area of compound)] found in the four Italian Goats' cheeses at the end of ripening. ISTD was nonadecanoic acid, methyl ester

Values are means (SD) for three batches of each type of cheese, analysed in duplicate

Compound name	Flor di Capra	Caprino di Cavalese	Caprino di Valsassina	Capritilla	Statistical significance§
	Relative Abundance to ISTD (ppb)				
<i>Acids</i>					
Butanoic acid	3308.2 <sup>ab</sup> (659.8)	7496.4 <sup>b</sup> (4859.8)	2.3 <sup>a</sup> (2.5)	301.9 <sup>a</sup> (348.6)	*
3-Methyl-butanoic acid	3280.1 <sup>a</sup> (737.1)	8905.9 <sup>a</sup> (5755.7)	44.8 <sup>a</sup> (49.5)	140.7 <sup>a</sup> (162.4)	**
2-Methyl-butanoic acid	666.6 <sup>a</sup> (328.3)	777.7 <sup>a</sup> (381.9)	587.8 <sup>a</sup> (306.7)	5437.3 <sup>b</sup> (2712.6)	*
Pentanoic acid	8.9 <sup>a</sup> (9.6)	55.7 <sup>a</sup> (38.7)	9213.5 <sup>a</sup> (4625.6)	7119.4 <sup>a</sup> (5436.2)	*
Hexanoic acid	6767.2 <sup>a</sup> (232.9)	93552.3 <sup>b</sup> (51903.6)	7363.4 <sup>a</sup> (1888.6)	7073.5 <sup>a</sup> (272.7)	*
2-Methyl-hexanoic acid	468.07 <sup>b</sup> (210.0)	45.1 <sup>a</sup> (42.1)	8.0 <sup>a</sup> (1.0)	1.9 <sup>a</sup> (0.9)	**
4-Methyl-hexanoic acid	10.1 <sup>a</sup> (10.5)	123.2 <sup>ab</sup> (60.5)	208.5 <sup>b</sup> (106.5)	10.0 <sup>a</sup> (1.9)	*
Heptanoic acid	0.4 <sup>a</sup> (0.3)	1602.2 <sup>b</sup> (746.6)	20.3 <sup>a</sup> (22.4)	2.3 <sup>a</sup> (1.0)	**
Octanoic acid	321.7 <sup>a</sup> (100.6)	119949.1 <sup>a</sup> (106409.6)	685264 <sup>b</sup> (303699.3)	791.7 <sup>a</sup> (97.9)	**
Nonanoic acid	9.1 <sup>a</sup> (0.4)	1499.1 <sup>b</sup> (731.5)	7072.4 <sup>c</sup> (923.2)	6.4 <sup>a</sup> (0.5)	*
Decanoic acid	1053.9 <sup>a</sup> (87.7)	105739.4 <sup>a</sup> (79408.5)	938621.8 <sup>b</sup> (226760.3)	1466.4 <sup>a</sup> (89.5)	***
Undecanoic acid	1619.1 <sup>a</sup> (1772.7)	354.5 <sup>a</sup> (96.5)	41608.8 <sup>b</sup> (13338)	185.2 <sup>a</sup> (16.5)	***
Dodecanoic acid	78.8 <sup>a</sup> (67.3)	234215.2 <sup>b</sup> (74771.1)	800596.9 <sup>c</sup> (114343)	4249.7 <sup>a</sup> (279.4)	***
Tridecanoic acid	113.1 <sup>a</sup> (10.9)	310.2 <sup>b</sup> (55.3)	597.6 <sup>c</sup> (9.4)	59.5 <sup>a</sup> (9.3)	***
Tetradecanoic acid	23845.3 <sup>a</sup> (2806.4)	186312.7 <sup>b</sup> (19852)	286348.6 <sup>c</sup> (7525.5)	47010.6 <sup>a</sup> (2692.8)	***
Pentadecanoic acid	22.0 <sup>a</sup> (6.7)	499.3 <sup>b</sup> (52.8)	687.8 <sup>c</sup> (37.6)	32.3 <sup>a</sup> (8.6)	***
n-Hexadecanoic acid	404.9 <sup>a</sup> (105.9)	5415.1 <sup>b</sup> (917.5)	8278.1 <sup>c</sup> (393.4)	1320.7 <sup>a</sup> (125.3)	***
11-Hexadecenoic acid (Z)	165.8 <sup>a</sup> (38.7)	153.1 <sup>a</sup> (34.8)	562.6 <sup>a</sup> (259.9)	282.2 <sup>a</sup> (73.2)	NS
Heptadecanoic acid	0.3 <sup>a</sup> (0.2)	17.9 <sup>b</sup> (6.3)	17.2 <sup>b</sup> (3.9)	0.8 <sup>a</sup> (0.5)	***
9-Octadecenoic acid (E)	63 <sup>a</sup> (9.7)	1084.1 <sup>b</sup> (215.7)	1779 <sup>c</sup> (240.3)	200 <sup>a</sup> (90.5)	***
Octadecanoic acid	6.8 <sup>a</sup> (5.7)	243.3 <sup>b</sup> (74.5)	123.2 <sup>ab</sup> (41.1)	25.9 <sup>a</sup> (6.1)	***
Total Acids	42213.4	768118.3	2789007	75692.4	
<i>Alcohols</i>					
2-Heptanol	256.6 <sup>a</sup> (279.2)	1326.8 <sup>a</sup> (931.6)	3128.5 <sup>a</sup> (1738.9)	1448.0 <sup>a</sup> (496.1)	NS
11-Dodecenol	429.9 <sup>a</sup> (439.3)	4573.2 <sup>a</sup> (3393.9)	12755.8 <sup>b</sup> (2426.3)	69.3 <sup>a</sup> (22.1)	***
3,7,11-Trimethyl-1-dodecanol	594 <sup>a</sup> (68.2)	5255.6 <sup>ab</sup> (1723.7)	2851.6 <sup>b</sup> (1617.9)	504.4 <sup>a</sup> (26.2)	**

Table 2. (Cont.)

Compound name	Flor di Capra	Caprino di Cavalese	Caprino di Valsassina	Capritilla	Statistical significance§
	Relative Abundance to ISTD (ppb)				
2-Methyl-2-buten-1-ol	186·8 <sup>c</sup> (21·1)	113 <sup>b</sup> (15·3)	59·3 <sup>a</sup> (10·5)	165·7 <sup>c</sup> (13·3)	***
4-Methyl-phenol	39·9 <sup>a</sup> (27·1)	757·1 <sup>b</sup> (85·4)	25·1 <sup>a</sup> (8·48)	11·4 <sup>a</sup> (3·1)	***
Phenylethyl alcohol	56 <sup>a</sup> (27·7)	208·7 <sup>b</sup> (60·1)	ND	30 <sup>a</sup> (11·9)	***
9-Octadecen-1-ol (E)	43·8 <sup>a</sup> (5·3)	96·7 <sup>a</sup> (59·6)	730·4 <sup>b</sup> (323·5)	39·1 <sup>a</sup> (2·2)	**
Total Alcohols	1607	12331·2	19550·8	2267·8	
<i>Aldehydes</i>					
Benzeneacetaldehyde	1472·6 <sup>a</sup> (1058·1)	1002 <sup>a</sup> (60·7)	1158·2 <sup>a</sup> (97·2)	579·2 <sup>a</sup> (38·8)	NS
Nonanal	191·5 <sup>a</sup> (72·2)	101·5 <sup>a</sup> (14·3)	2653 <sup>b</sup> (1724·7)	167·4 <sup>a</sup> (53·5)	*
Hexadecanal	606·2 <sup>cb</sup> (38·1)	511·1 <sup>b</sup> (41·8)	658·4 <sup>c</sup> (29·4)	395·1 <sup>a</sup> (23·2)	***
13-Octadecenal (Z)	127·9 <sup>a</sup> (48·9)	70·7 <sup>a</sup> (21·7)	52·2 <sup>a</sup> (16·7)	130·2 <sup>a</sup> (23)	NS
Total Aldehydes	2398·3	1685·3	4521·9	1271·9	
<i>Amines</i>					
4-Methoxy benzenamine	2650·2 <sup>ab</sup> (1093·7)	1062·92 <sup>ab</sup> (461·1)	ND	4661·39 <sup>b</sup> (1949·1)	*
Total Amines	2650·2	1062·9	ND	4661·4	
<i>Aromatic Compounds</i>					
Indole	6·8 <sup>a</sup> (1·4)	475·9 <sup>c</sup> (110·5)	49·1 <sup>ab</sup> (10·1)	232·8 <sup>b</sup> (28·2)	***
alpha-Caryophyllene	1·1 <sup>a</sup> (0·5)	88·3 <sup>a</sup> (34)	83·9 <sup>a</sup> (83·7)	1·7 <sup>a</sup> (0·62)	NS
Total Aromatic Compounds	7·9	564·3	133	234·5	
<i>Esters</i>					
3-Methyl butanoate	425·6 <sup>a</sup> (245·3)	2041·4 <sup>a</sup> (998·2)	25172 <sup>a</sup> (18742)	9718·7 <sup>a</sup> (4076·3)	NS
Ethyl-butanoate	353 <sup>b</sup> (55·5)	169·11 <sup>a</sup> (22·5)	400·69 <sup>b</sup> (42·4)	284·25 <sup>ab</sup> (36·2)	**
Ethyl-decanoate	150·4 <sup>a</sup> (38·4)	2887 <sup>d</sup> (183·6)	1588·6 <sup>c</sup> (102·2)	1069·2 <sup>b</sup> (53·2)	***
Ethyl-hexanoate	63·4 <sup>a</sup> (61·1)	853·5 <sup>c</sup> (90·1)	583·6 <sup>b</sup> (67·9)	182 <sup>a</sup> (13·8)	***
Cyclopentyl-hexanoate	6·7 <sup>a</sup> (7·6)	392·8 <sup>b</sup> (46·3)	126·1 <sup>a</sup> (139·3)	ND	*
Isopentyl-hexanoate	811·9 <sup>a</sup> (922·4)	607·5 <sup>a</sup> (189·7)	549·6 <sup>a</sup> (421·3)	11·1 <sup>a</sup> (0·6)	NS
Methyl-decanoate	5·3 <sup>a</sup> (1·5)	75·7 <sup>ab</sup> (2·9)	105·1 <sup>b</sup> (59·3)	1·7 <sup>a</sup> (0·6)	*
2-Butyl-octanoate	ND	392·2 <sup>b</sup> (47·7)	1·1 <sup>a</sup> (0·6)	ND	***
Propyl-Decanoate	0·3 <sup>a</sup> (0·4)	79·4 <sup>b</sup> (11·6)	3·8 <sup>a</sup> (1·8)	1·9 <sup>a</sup> (0·6)	***

Table 2. (Cont.)

Compound name	Flor di Capra	Caprino di Cavalese	Caprino di Valsassina	Capritilla	Statistical significance§
	Relative Abundance to ISTD (ppb)				
Isobutyl-caproate	4.2 <sup>a</sup> (1.2)	948.1 <sup>c</sup> (87.5)	406.7 <sup>b</sup> (214.3)	5.2 <sup>a</sup> (0.8)	***
Butyl-Azelaaldehydate	1.3 <sup>a</sup> (1.4)	1325.1 <sup>b</sup> (179.7)	11.4 <sup>a</sup> (1)	1 <sup>a</sup> (0.4)	***
3-Methylbutyl-pentadecanoate	7 <sup>a</sup> (1.1)	138.9 <sup>b</sup> (19.3)	16.8 <sup>a</sup> (2.5)	2.9 <sup>a</sup> (0.8)	***
Dodecanoic acid, 1-methylpropyl ester	21.8 <sup>a</sup> (25.2)	431.7 <sup>b</sup> (61.4)	1.7 <sup>a</sup> (0.7)	0.4 <sup>a</sup> (0.3)	***
Ethyl-tetradecanoate	27.6 <sup>a</sup> (4.18)	673.08 <sup>c</sup> (52.07)	393.59 <sup>b</sup> (15.61)	1078.27 <sup>d</sup> (67.20)	***
2-Tridecyl-valerate	55.8 <sup>a</sup> (7.7)	118.6 <sup>b</sup> (7)	154.7 <sup>c</sup> (5.5)	67.2 <sup>a</sup> (3.8)	***
Ethyl-hexadecanoate	27.3 <sup>a</sup> (21.8)	225.4 <sup>c</sup> (21.9)	114.4 <sup>b</sup> (2.9)	508.4 <sup>d</sup> (31.4)	***
Z-10-Tetradecen-1-ol acetate	38.4 <sup>b</sup> (6.6)	31.5 <sup>ab</sup> (6.7)	35.6 <sup>ab</sup> (3.4)	20.5 <sup>a</sup> (2)	*
Total Esters	2000.1	11391	29665.5	12952.7	
<i>Ketones</i>					
2-Heptanone	2824.8 <sup>a</sup> (245.8)	2871.1 <sup>a</sup> (570.6)	10172.7 <sup>b</sup> (3071)	4347.6 <sup>a</sup> (1095.5)	**
2-Nonanone	1585.2 <sup>a</sup> (139.3)	1913.3 <sup>a</sup> (190.4)	1781.1 <sup>a</sup> (231.8)	1702.7 <sup>a</sup> (58)	NS
2-Undecanone	571.4 <sup>a</sup> (42)	800.6 <sup>b</sup> (18.1)	790 <sup>b</sup> (127.5)	601.1 <sup>a</sup> (21.9)	***
2-Tridecanone	910.7 <sup>a</sup> (99.8)	1155.2 <sup>a</sup> (64.1)	896.2 <sup>a</sup> (91.7)	938.4 <sup>a</sup> (44.8)	NS
2-Pentadecanone	1673.6 <sup>a</sup> (330.3)	1820.5 <sup>a</sup> (117.9)	1385.8 <sup>a</sup> (58.3)	1538.3 <sup>a</sup> (94.1)	NS
Acetophenone	8.9 <sup>a</sup> (1.1)	101.2 <sup>b</sup> (27.5)	10.3 <sup>a</sup> (1.2)	52.3 <sup>a</sup> (4.3)	***
2-Ethyl-cyclohexanone	29.4 <sup>a</sup> (6.8)	89.4 <sup>a</sup> (33.6)	119.8 <sup>a</sup> (61.7)	23.4 <sup>a</sup> (1.5)	NS
Total Ketones	7603.9	8751.3	15155.8	9203.8	
<i>Lactones</i>					
gamma Nonalactone	4273.4 <sup>c</sup> (539.4)	1647.6 <sup>a</sup> (145.9)	2854.2 <sup>ab</sup> (558.5)	2971.9 <sup>bc</sup> (159.5)	***
gamma Decalactone	74.5 <sup>a</sup> (8.1)	198.5 <sup>a</sup> (52.6)	1231.4 <sup>b</sup> (408.2)	107.1 <sup>a</sup> (6)	***
delta Decenolactone	2970.5 <sup>ab</sup> (372.9)	3594.4 <sup>a</sup> (596.9)	13823.8 <sup>c</sup> (5416.6)	3848.4 <sup>bc</sup> (219.3)	**
delta Decalactone	775.1 <sup>ab</sup> (92.4)	688.3 <sup>a</sup> (76.2)	1063.1 <sup>c</sup> (74.6)	940.4 <sup>bc</sup> (50.3)	**
4-Hydroxy-6-dodecenoic acid lactone (Z)	1842.4 <sup>b</sup> (240.6)	927 <sup>a</sup> (156.3)	2264.6 <sup>b</sup> (272.3)	806.8 <sup>a</sup> (45.7)	***
delta Undecalactone	4275.3 <sup>c</sup> (536.7)	1684.7 <sup>a</sup> (223.4)	2255.1 <sup>ab</sup> (455.2)	2970.7 <sup>b</sup> (159.9)	***
delta Dodecalactone	1128 <sup>a</sup> (104.4)	1233.2 <sup>ab</sup> (65.3)	1410.3 <sup>b</sup> (67.2)	1211.7 <sup>ab</sup> (76.2)	*



**Table 2.** (Cont.)

Compound name	Flor di Capra	Caprino di Cavalese	Caprino di Valsassina	Capritilla	Statistical significance§
	Relative Abundance to ISTD (ppb)				
Tetrahydro-6-octyl-(2H)-pyran-2-one	574.4 <sup>a</sup> (68.4)	760.7 <sup>b</sup> (42.6)	987 <sup>c</sup> (32.5)	605.5 <sup>a</sup> (35.9)	***
gamma Palmitolactone	52.5 <sup>a</sup> (6.1)	113.5 <sup>b</sup> (8.2)	157.7 <sup>c</sup> (5)	48.6 <sup>a</sup> (2.2)	***
Total Lactones	15966.2	10847.9	26047.2	13511.1	
<i>Miscellaneous</i>					
Phytol	57.3 <sup>a</sup> (14.1)	72.5 <sup>a</sup> (13.9)	172.3 <sup>b</sup> (48.4)	59.3 <sup>a</sup> (6.9)	**
Total Miscellaneous	57.3	72.5	172.3	59.3	
<i>Sulphur compounds</i>					
5-(2-propenyl)-2(5H)-Thiophenone	134.3 <sup>a</sup> (13)	216.9 <sup>b</sup> (7.2)	192.8 <sup>b</sup> (8.2)	157.1 <sup>a</sup> (7.9)	***
3-(methylthio)-Propanal	12.7 <sup>a</sup> (8)	122.1 <sup>b</sup> (36.1)	20.2 <sup>a</sup> (5.3)	1.9 <sup>a</sup> (1.2)	***
Total Sulphur compounds	147	339	213	59	

<sup>a-d</sup> Means within a row with different superscript letters are significantly different at the *P* value in the last column.

§ Statistical significance between the four Italian goats' cheeses: \*\*\* *P*<0.001; \*\* *P*<0.01; \* *P*<0.05; NS not significant (*P*>0.05).

ND, not detected.

The variability of the volatile components among the three batches of each type was not significant (*P*>0.05).

**Table 3.** Sensory profiles of the four Italian goats' cheeses at the end of ripening

Values are means for three batches of each type of cheese, analyzed in duplicate. Intensities were scored on a 15-point universal intensity scale using the Spectrum™ method

Flavour attribute	Flor di Capra	Caprino di Cavalese	Caprino di Valsassina	Capritilla
Cooked/milky	2.2 <sup>a</sup>	1.2 <sup>b</sup>	0.5 <sup>c</sup>	1.1 <sup>b</sup>
Whey	2.8	ND§	ND	ND
Diacetyl	1.3	ND	ND	ND
Milkfat	3.2 <sup>a</sup>	1.5 <sup>c</sup>	1.0 <sup>c</sup>	2.3 <sup>b</sup>
Fruity	ND	1.3 <sup>b</sup>	2.6 <sup>a</sup>	0.6 <sup>c</sup>
Sulphur	2.3	ND	ND	2.8
Free fatty acid	ND	1.3 <sup>b</sup>	4.3 <sup>a</sup>	1.6 <sup>b</sup>
Brothy	ND	3.4 <sup>a</sup>	2.8 <sup>b</sup>	3.3 <sup>a</sup>
Nutty	1.0 <sup>b</sup>	2.3 <sup>a</sup>	2.2 <sup>a</sup>	2.3 <sup>a</sup>
Waxy/animal	3.2 <sup>a</sup>	1.8 <sup>c</sup>	1.3 <sup>d</sup>	2.7 <sup>b</sup>
Cow/phenolic	ND	3.8	ND	ND
Amine/proteolytic	ND	ND	ND	3.9
Sweet	2.2 <sup>c</sup>	2.3 <sup>c</sup>	2.8 <sup>b</sup>	3.2 <sup>a</sup>
Sour	2.8 <sup>a</sup>	2.0 <sup>b</sup>	2.5 <sup>a</sup>	2.5 <sup>a</sup>
Salty	3.0 <sup>c</sup>	4.2 <sup>a</sup>	4.3 <sup>a</sup>	3.8 <sup>b</sup>
Bitter	ND	ND	ND	1.3
Burn	ND	ND	1.8	ND

<sup>a-d</sup> Means within a row with different superscript letters are significantly different (*P*<0.05).

§ ND, not detected.

characterization permitted cheeses to be differentiated and to establish relationships between technology, biochemical process and sensory attributes.

## References

- Caponio F, Pasqualone A & Gomes T** 2001 Apulian Cacioricotta goat's cheese: technical interventions for improving yield and organoleptic characteristics. *European Food and Research Technology* **213** 17–182
- Carunchia-Whetstine ME, Karagul-Yuceer Y, Avsar YK & Drake MA** 2003 Identification and quantification of character aroma components in fresh Chevre-style goat cheese. *Journal of Food Science* **68** 2441–2447
- Casalta E, Noël Y, Le Bars D, Carré C, Achilleos C & Maroselli MX** 2001 Caractérisation du fromage Bastelicaccia. *Lait* **81** 529–546
- Coda R, Brechany E, De Angelis M, De Candia S, Di Cagno R & Gobbetti M** 2006 Comparison of the compositional, microbiological, biochemical, and volatile profile characteristics of nine Italian ewes' milk cheeses. *Journal of Dairy Science* **89** 4126–4143
- Deckelbaum RJ, Hamilton JA, Moser A & Bengtsson-Olivecrona T** 1990 Medium-chain vs long-chain triacylglycerol emulsion hydrolysis by lipoprotein lipase and hepatic lipase: Implications for the mechanism of lipase action. *Biochemistry* **29** 1136–1142
- Di Cagno R, Banks J, Sheehan J, Fox PF, Brechany EY, Corsetti A & Gobbetti M** 2003 Comparison of the microbiological, compositional, biochemical, volatile profile and sensory characteristics of three Italian PDO ewes' milk cheeses. *International Dairy Journal* **13** 961–972
- Drake MA, McIngvale S, Gerard PD, CadWallader KD & Civile GV** 2001 Development of a descriptive language for Cheddar cheese. *Journal of Food Science* **66** 1422–1427

- FAOSTAT** 2004 FAO Agricultural Statistics. Food And Agriculture Organization of the United Nations. Rome, Italy (<http://faostat.fao.org>)
- Fontecha J, Peláez C, Juárez M, Requena T & Gómez C** 1990 Biochemical and microbiological characteristics of artisanal hard goat's cheese. *Journal of Dairy Science* **73** 1150–1157
- Fox PF, McSweeney PLH & Lynch CM** 1998 Significance of non-starter lactic acid bacteria in Cheddar cheese. *Australian Journal of Dairy Technology* **53** 5383–5389
- Franco I, Prieto B, González J & Carballo J** 2001 The Spanish traditional goat's milk cheeses: a review. *Alimentaria* **319** 63–82
- Fresno JM, Tornadijo ME, Carballo J, Bernardo A & González-Prieto J** 1997 Proteolytic and lipolytic changes during the ripening of a Spanish craft goat cheese (Armada variety). *Journal of the Science of Food and Agriculture* **75** 148–154
- Gobbetti M, Folkertsma B, Fox PF, Corsetti A, Smacchi E, De Angelis M, Rossi J, Kilcawley K & Cortini M** 1999 Microbiology and Biochemistry of Fossa (pit) cheese. *International Dairy Journal* **9** 763–773
- Gomez-Ruiz JA, Ballesteros C, González Viñas MA, Cabezas L & Martínez-Castro I** 2002 Relationships between volatile compounds and odour in Manchego cheese: comparison between artisanal and industrial cheeses at different ripening times. *Lait* **82** 613–628
- Guillen MD, Ibargoitia ML, Sopelana P & Palencia G** 2004a Components detected by headspace-solid phase microextraction in artisanal fresh goat's cheese smoked using dry prickly pear (*Opuntia ficus indica*). *Lait* **84** 385–397
- Guillen MD, Ibargoitia ML, Sopelana P, Palencia G & Fresno M** 2004b Components detected by means of solid-microextraction and gas-chromatography/mass spectrometry in the headspace of artisan fresh goat cheese smoked by traditional methods. *Journal of Dairy Science* **87** 284–299
- Juárez M** 1986 Physico-chemical characteristics of goat's milk as distinct from those of cow's milk. *International Dairy Federation Bulletin* no. **202** 54–67
- Karagul-Yuceer Y, Drake MA & Cadwallader KR** 2001 Aroma-active components of nonfat dry milk. *Journal of Agricultural Food Chemistry* **49** 2948–2953
- Larrayoz P, Addis M, Gauch R & Bosset JO** 2001 Comparison of dynamic headspace and simultaneous distillation extraction techniques used for the analysis of the volatile components in three European PDO ewes' milk cheeses. *International Dairy Journal* **11** 911–926
- Le Quéré J-L, Pierre A, Riaublanc A & Demazières D** 1998 Characterization of aroma compounds in the volatile fraction of soft goat cheese during ripening. *Lait* **78** 279–290
- Mas M, Tabla R, Moriche J, Roa I, González J, Rebollo JE & Cáceres P** 2002 Iborea goat's milk cheese: Microbiological and physicochemical changes throughout ripening. *Lait* **82** 579–587
- Medina M & Nuñez M** 2004 Cheeses made from ewes' and goats milk. In *Cheese: Chemistry, Physics and Microbiology*, pp 279–299 (Eds PF Fox, PLH McSweeney, TM Cogan & TP Guinee). Chapman and Hall: London
- Morgan F & Gaborit P** 2001 The typical flavour of goat milk products: technological aspects. *International Journal of Dairy and Technology* **54** 38–40
- Poveda JM & Cabezas L** 2006 Free fatty acids composition of regionally-produced Spanish goat cheese and relationship with sensory characteristics. *Food Chemistry* **95** 307–311
- Preininger M & Grosch W** 1994 Evaluation of key odorants of the neutral volatiles of Emmentaler cheese by the calculation of odour activity values. *Lebensmittel-Wissenschaft und-Technologie* **27** 237–244
- Pripp A, Shakeel-Ur-Rehman H, McSweeney PLH & Fox PF** 1999 Multivariate statistical analysis of peptide profiles and free amino acids to evaluate effects of single strain starters on proteolysis in miniature Cheddar-type cheeses. *International Dairy Journal* **9** 473–479
- Salles C, Hervé C, Septier C, Deimazières D, Lesschaeve I, Issanchou S & Le Quéré JL** 2000 Evaluation of taste compounds in water-soluble extract of goat cheese. *Food Chemistry* **68** 429–435
- Sánchez I, Seseña S, Poveda JM, Cabezas L & Palop L** 2005 Phenotypic and genotypic characterization of lactobacilli isolated from Spanish goat cheeses. *International Journal of Food Microbiology* **102** 355–362
- Suzzi G, Caruso M, Gardini F, Lombardi A, Vannini L, Guerzoni ME, Andrighetto C & Lanorte MT** 2000 A survey of the enterococci isolated from an artisanal Italian goat's cheese (semicotto caprino). *Journal of Applied Microbiology* **89** 267–274