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## Ripening changes of the chemical composition, proteolysis, volatile fraction and organoleptic characteristics of a white-brined goat milk cheese

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#### ABSTRACT

In this study, a white-brined cheese made from goat milk (from the Greek native breed *Capra prisca*) was manufactured using the technology based on Feta cheese-making. The composition, proteolysis, volatiles and sensory properties of this cheese were studied at different ripening days. No significant differences were found for the main physicochemical parameters of the cheese during ripening and storage. Retinol and  $\alpha$ -tocopherol, the fat-soluble vitamins decreased after 60 d of ripening. Proteolysis, as assessed by measuring the levels of soluble nitrogen, increased with time. Free fatty acids were the most abundant group of chemical compounds found in the cheese. Most of the volatiles (apart from free fatty acids) reached their highest levels after 60 days of ripening. The cheese received satisfactory scores during organoleptic evaluation.

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#### 1. Introduction

The national agricultural economy of many countries in the Mediterranean basin (especially Greece, France and Spain) depends on goat population. According to FAOSTAT (2015), the world's goat population increased by around 69% between 1991 and 2014, while, the number of cattle grew by 14% and sheep by about 2%. The production of goat milk increased by around 80% between 1991 and 2013 (FAOSTAT, 2015), pointing to a promising future for this sector, therefore any effort to encourage production and scientific research in this field will be positive for the industry and for the quality of goat milk and its products. In 2014, Greece had 4.25 millions goat heads; Spain 2.70 millions heads and France 1.27 millions goat heads. Also in the same year, goat milk production in France, Spain and Greece was as high as 604, 447 and 351 million L, respectively (EUROSTAT, 2016).

The native breed *Capra prisca* constitutes the major part (85%) of goat population in Greece. It is durable, lives generally in small flocks under extensive breeding systems and is very well adapted to the climate and the mountainous conditions (Simos et al., 1991;

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http://dx.doi.org/10.1016/j.smallrumres.2016.10.022 0921-4488/© 2016 Elsevier B.V. All rights reserved. Kondyli et al., 2007). Moatsou et al. (2006, 2007) found that goat milk from indigenous Greek breeds has high casein content, resulting from the abundance of "strong"  $\alpha_{s1}$ -casein variants and high fat content.

Goat milk has gained economic importance via its utilization in cheesemaking, in the Mediterranean and many eastern European countries. In the European Union, goat cheeses have gained popularity due to the increased interest of consumers in both the traditional cheesemaking practices and the sensorial and nutritional value attributed to goat milk. Ripened goat cheeses are characterized by a piquant and peppery-sharp flavour. In Greece, goat milk is mainly used for the cheesemaking of Feta cheese, which is a Protected Denomination of Origin (PDO) product. According to Commission Regulation EC 1829 (2002) a quantity of goat milk up to 30% can be added to the sheep milk in the manufacture of Feta cheese. Goat milk is also used for the manufacture of various PDO cheeses and other cheese varieties which are produced in limited scale (Pappa et al., 2006a,b; Mallatou et al., 1994).

White brined goat milk cheeses, in Greece, were mainly produced in small scale. Nowadays they become more and more popular and are produced by following almost the similar to Feta procedure. In general, there is little information about white brined goat cheeses (Salameh et al., 2016; Barac et al., 2016, 2013; Moatsou and Govaris 2011; Asteri et al., 2010; Pappa and Sotirakoglou,







2009; Alichanidis and Polychroniadou, 2008; Tzanetakis et al., 1995; Litopoulou-Tzanetaki and Tzanetakis, 1992). Research on their technological aspects and ripening biochemistry, especially on their flavour chemistry, will enhance our understanding of the role of specific compounds contributing to cheese flavour and relate cheese flavour to the technology of cheese production. Therefore, the aim of the present work was to study the composition, proteolysis, sensory properties and volatile compounds of a white brined cheese made by the goat milk of the indigenous Greek breed, during ripening, as these results could help the cheese processors to produce a high quality white-brined cheese from goat milk of the indigenous breed of *Capra prisca*.

#### 2. Materials and methods

#### 2.1. Cheese manufacture

Three cheesemaking trials were carried out in the pilot plant of Dairy Research Department using 50 kg goat milk according to the following procedure, which is based on the Feta cheese manufacture.

The fresh raw goat milk used for the cheese manufacture, was from a farm which bred 300 goats of the native goat breed (*Capra prisca*) and was located in the semi mountainous (600–800 m altitude) region of Ioannina, North Western Greece. Cheesemakings were carried out in early summer when goats were fed exclusively by grazing in semi-mountainous and mountainous pastures.

Goat milk was pasteurised at 63 °C for 30 min in a double walled stainless steel vat and cooled to 35 °C. After cooling (35 °C) the normal starter culture (Streptococcus salivarius subsp. thermophilus and Lactobacillus delbrueckii subsp. bulgaricus 1:1) at a rate of 7.5 g kg<sup>-1</sup> was added and allowed to rest for 30 min. Then CaCl<sub>2</sub> solution (10% w/v) at a rate of 100 mL/100 kg milk and powdered calf rennet (HA-LA, Hansen's Laboratorium, Copenhagen, Denmark) were added to achieve curdling in approximately 50 min. The cheese curd was cut into small cubes 2.5 cm side, allowed to rest for 10 min and transferred into rectangular moulds. After draining the curd was cut into blocks weighing about 1 kg and put into tin vessels. Granular recrystallised NaCl equivalent to 25.0 g kg<sup>-1</sup> of cheese was added and after one day the drained whey was removed and replaced by  $70.0 \,\mathrm{g \, kg^{-1}}$ NaCl solution in a ratio of brine volume to cheese weight of 1:4. The tin vessels were sealed and left for ripening at 16-18 °C for approximately 15 days until the pH and moisture were lower than 4.6 and 56% respectively. Subsequently the cheeses were transferred into the cold storage rooms  $(3-4^{\circ}C)$  and remained there for up to six months.

Cheese samples were analysed for their composition and proteolysis at 2, 20, 60, 120 and 180 days and volatile compounds at 60 and 180 days of age.

#### 2.2. Chemical analyses

Cheese samples were analysed for moisture, salt, fat and protein content, pH, total nitrogen (TN) and soluble nitrogen (SN) fractions, i.e., water-soluble nitrogen (WSN), nitrogen soluble in 12% trichloroacetic acid (TCA-N) or in 5% phosphotungstic acid (PTA-N), as described by Katsiari et al. (2002).

The fat soluble vitamins,  $\alpha$ -tocopherol (vitamin E) and alltrans-retinol (vitamin A) were determined according to Panfilli (1994) with modifications of the liquid chromatographic conditions described by Kondyli et al. (2007).

The volatile compounds of cheese samples at 60 and 180 days of storage were studied by SPME-GC–MS analysis. Homogenized cheese samples (1.0g each) were placed in 10 mL glass vials fitted with a Teflon – lined septum sealed with an aluminum crimp seal through which an SPME syringe needle, equipped with a 2 cm fibre coated with 50–30 nm divinylbenzene-carboxen on dimethyl siloxane (DVB/CAR PDMS) bonded to a flexible fused silica core (Supelco, Bellefonte, PA, USA) was introduced. The fibre coating was exposed for 50 min at 60  $^{\circ}$ C to the cheese headspace under shaking in an automatic SPME autosampler (AOC 5000 Auto Injector, PAL, CTC Analytics, Zwingen, Switzerland). The absorbed volatile compounds were then analysed by GC-MS (GCMSQP2010, Shimadzu, Tokyo, Japan, capillary column Supelco CO Wax-10, 30 m length, 0.32 mm inside diameter and 0.50 µm film thickness). Helium was used as the carrier gas (linear velocity 0.8 mLmin<sup>-1</sup>). The injector was operated at 260 °C and the oven was set to 45 °C for 5 min, then at a rate of  $10 \,^{\circ}$ C min<sup>-1</sup> to  $80^{\circ}$  and finally at a rate of  $5 \,^{\circ}$ C min<sup>-1</sup> to 240° for 10 min. The mass selective detector (QP2010, Shimadzu) operated in the electron impact mode with 70 eV electron energy and interface temperature 270 °C. Identification was done by comparison with standard compounds, retention indices (RI) and data obtained by NIST05 library (Gaithersburg, MD, USA). Semi-quantification was performed by integrating the peak areas of total ion chromatograms (TIC) by the Shimadzu GCMS Solution software

#### 2.3. Organoleptic (sensory) evaluation

Samples of cheese were cut into small cubes 2 cm side and placed on white plates. The pieces were tempered by holding at ambient temperature  $(18 \pm 2 \,^{\circ}C)$  and presented to the panellists. The cheese was evaluated by a five member panel trained and familiar to this type of cheeses, after 60, 120 and 180 days of storage. Also the panellists instructed to report any defects of the cheese in appearance (e.g. cracks, unusual colour etc), body and texture (e.g. hard, soft, crumbly, spongy etc) and flavour (e.g. acid, rancid, bitter etc) detected according to the IDF (1987) guide for sensory evaluation of cheese.

#### 2.4. Statistical analysis

The data were analysed by one way analysis of variance (ANOVA) performed using the software Statgraphics (Statistical Graphics Corp., Rockville, MD, USA). When significant (P < 0.05) differences were found during storage, means were separated by Least Significance Difference test (LSD).

#### 3. Results and discussion

#### 3.1. Physico-chemical characteristics of cheeses

The composition of the white-brined goat milk cheese during ripening and storage is shown in Table 1. No significant (P<0.05) differences were found for moisture, fat, pH and protein contents of cheese during ripening and storage. Statistical analysis showed that the NaCl and the salt in moisture (S/M) content of goat whitebrined cheeses were increasing until transfer to the storage room because at the very early stages of ripening no salt equilibrium between the cheese mass and brine was reached. It remained stable thereafter, without significant differences (P>0.05). The S/M content of a cheese defines the feasibility of the development of microorganisms and the kinetics of the enzymatic reactions during ripening (Guamis et al., 1997). Cheese of the present study fulfilled the requirements of the Greek market for first quality white-brined cheese, that is  $\leq$ 56% moisture and  $\geq$ 43% fat in dry matter (Greek Codex Alimentarius, 2009). In general, similar results were found in Teleme cheese (Pappa et al., 2006a,b), Feta-type cheese (Mallatou et al., 1994) and pickled cheese (Tzanetakis et al., 1995) made from goat milk.

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