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Lipolysis of cheeses made from goat milk treated by ultra-high pressure homogenization

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ABSTRACT

The effect of ultra high-pressure homogenization of goat milk (200 MPa, 30 °C) on the lipolytic profile of cheese was studied and compared to cheeses made from homogenized–pasteurized (18 + 2 MPa, 72 °C for 15 s) and pasteurized (72 °C, 15 s) milk. Cheeses produced from ultra high-pressure homogenized milk presented similar levels of lipolysis to those made from pasteurized milk; however, the former showed higher contents of short-chain fatty acids at early stages of ripening. Cheeses made from homogenized–pasteurized milk exhibited the highest levels of lipolysis, presenting twice the levels of total free fatty acids at 30 and 60 days of ripening. Differences in the lipolysis level between the two homogenization technologies could be caused by different changes produced on milk fat globules, making easier or more difficult the fat accessibility for the lipolytic enzymes present in cheese.

1. Introduction

Lipolysis is an important biochemical event which takes place during the ripening of many cheese varieties, especially in mould-ripened cheeses and cheeses from goat and sheep milk. Lipolysis releases free fatty acids (FFA) which contribute directly to cheese flavour, especially short- and medium-chain FFA. In addition, FFA also act as substrates for further reactions producing highly flavoured catabolic end products (Collins, McSweeney, & Wilkinson, 2003). Nevertheless, extensive lipolysis may be considered undesirable in most cheese varieties (Sousa & Malcata, 1997).

The lipolysis in cheese could be catalysed by lipases from native milk lipoprotein lipase (LPL), rennet preparations, starter lactic acid bacteria (LAB), non-starter LAB, adjunct cultures or secondary mould cultures, and exogenous lipolytic enzymes (Collins et al., 2003). FFA can also be produced from the metabolism of carbohydrates and amino acids by bacteria (Fox & Wallace, 1997; Urbach, 1993).

The indigenous LPL, is nearly completely inactivated by HTST pasteurization (Beuvier & Buchin, 2004), although the inactivation varies according to the severity of the heat treatment. On the other hand, commercial calf and bovine liquid rennets are normally free from lipolytic activity (Georgala et al. 2005). The effect of cheese microflora on lipolysis occurs via the esterase/lipase systems of lactic and propionic acid bacteria, non-starter LAB, surface micro-organisms, yeast and moulds (McSweeney & Sousa, 2000). LAB, especially *Lactococcus* and *Lactobacillus* spp. are weakly lipolytic (Fox & Stepaniak, 1993), however, they are responsible for the liberation of significant levels of FFA in many cheese varieties because of the high populations reached by these genera.

Ultra high-pressure homogenization (UHPH) is one of the food preservation treatments that is being developed and applied as a minimal process for the production of a wide variety of safe and nutritious foods. The principle of the operation is similar to that of conventional homogenizers used in the dairy industry except that it works at higher pressures (up to 400 MPa). UHPH is being studied for the treatment of milk in order to inactivate microorganisms, to produce very fine and stable emulsions and to modify the rheological and/or the coagulation properties of milk and dairy products.

The possibility of affecting the particle size distribution of fat globules, protein structure, and enzyme activity, has stimulated numerous studies on dairy products. Several authors have described the possible application of UHPH in the processing of milk (Datta, Hayes, Deeth, & Kelly, 2005; Hayes, Fox, & Kelly, 2005; Pereda, Ferragut, Buffa, Guamis, & Trujillo, 2008), for the production of yoghourt (Serra, Trujillo, Pereda, Guamis, & Ferragut, 2008; Serra, Trujillo, Guamis, & Ferragut, 2009a, 2009b, 2009c), dairy

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fermented beverages (Masson, Rosenthal, Calado, Deliza, & Tashima, 2011) and cheese (Escobar et al., 2011; Zamora, Ferragut, Juan, Guamis, & Trujillo, 2011). Also, the effects of UHPH on milk enzymes activity (Hayes & Kelly, 2003) and coagulation properties of milk have been described (Lodaite, Chevalier, Armaforte, & Kelly, 2009; Sandra & Dalgleish, 2007; Zamora, Ferragut, Jaramillo, Guamis, & Trujillo, 2007). UHPH technology (300 MPa, inlet temperature of 30 °C) is able to produce milk with microbiological characteristics similar to high pasteurized (90 °C, 15 s) milk (Pereda, Ferragut, Quevedo, Guamis, & Trujillo, 2007), while preserving its ability to be coagulated by rennet (Zamora et al., 2007). UHPH promotes greater reduction of fat globule size than conventional homogenization (Zamora et al., 2007), which would also lead to a greater potential for lipolysis to occur.

The objective of the present study was to quantify the release of FFA in goats' milk cheeses made from UHPH milk (UH-cheeses) in order to assess the effect of this technology on cheese lipolysis. The results obtained were compared with those from cheeses produced by conventional homogenized–pasteurized (18 + 2 MPa, 72 °C for 15 s, PH-cheeses) and heat-pasteurized (72 °C for 15 s, PA-cheeses) milks.

2. Material & methods

2.1. Milk treatments

Milk was obtained from a herd of Murciano-Granadina goats (Valencia, Spain). The milk batch was divided into three portions, and each of these portions was used for cheese making following the same protocol. A portion of the cheese milk was pasteurized (PA) at 72 °C for 15 s with a Finamat heat exchanger (model 6500/010, GEA Finnah GmbH, Ahaus, Germany) and another (PH) was homogenized at 18 + 2 MPa with a Niro Soavi homogenizer (model X68P Matr. 2123, Niro Soavi, Parma, Italy) and then pasteurized at 72 °C for 15 s. Finally, the third portion of milk was submitted to a single-pass UHPH treatment of 200 MPa on the primary homogenizing valve using a Stansted high pressure homogenizer (model FPG11300:400, Stansted Fluid Power Ltd., Essex, UK) at an inlet temperature of 30 ± 1 °C. The flow rate of milk in the homogenizer was approximately 120 h^{-1}. To minimize temperature retention after treatment, 2 spiral-type heat-exchangers (Garvía, Barcelona, Spain) located behind the cooling system were used. Milk temperature reached at valve level throughout the treatment was 82.75 ± 0.96 °C, with very short time retention (≤0.7 s), and the final temperature was ≤25 °C. Three independent productions of cheese were carried out within an interval of a month. UHPH conditions used in this work were selected from the results obtained in a previous study based on the microbiological and rennet coagulation properties of goat milk treated by UHPH (Trujillo, Zamora, Pereda, Quevedo, & Guamis, 2008).

2.2. Cheese making

Milk was heated to 32 °C, and a 2 L/100 L of lactic starter (*Lactococcus lactis* spp. *lactis* and *L. lactis* spp. *cremoris*, Choozit MA 11 Lyo, Danisco, France) was added. Subsequently, 25 ml/100 L milk of $CaCl_2$ and 20 ml/100 L of calf rennet (520 mg chymosin L^{-1}, Laboratories Arroyo, Santander, Spain) were added. After the coagulation, the curd was cut, drained, moulded (14.5 cm i.d. × 8.5 cm height) into pieces of about 1300 g, and pressed in a pneumatic press (Garvia S.A., Barcelona, Spain) at 0.5 kPa for 1 h and completed at 2 kPa for 14 h and salted by immersion in brine (19% NaCl w/w) for 4 h. Cheeses were ripened in a room at 14 °C and 85% relative humidity. Samples were taken periodically at 1, 15, 30 and 60 days of ripening.

2.3. Compositional analysis

Cheeses were analysed for moisture content and fat according to IDF (1982) and Van Gulik methods (ISO, 1975), respectively.

2.4. Free fatty acid profile analysis

FFA were extracted according to the modified method of De Jong and Badings (1990) as described by Juan, Ferragut, Buffa, Guamis, and Trujillo (2007). Gas chromatography was carried out with a Hewlett Packard chromatograph (Wilmington, DL, USA) equipped with a flame ionization detector at 300 °C, using a fused silica capillary column DB-FFAP (30 m × 0.32 × 0.25 μm, Agilent, Santa Clara, CA, USA) protected by a non polar guard column (5 m × 0.32 mm, Teknokroma, Barcelona, Spain). The carrier gas was high purity helium at flow rates of 0.9 ml min^{-1}. Temperature was raised from 75 to 200 °C at 5 °C min^{-1}, then was heated at 220 at 2 °C min^{-1}, then at 230 °C at 1 °C min^{-1} and finally held at 240 °C for 6 min. The output signal from the detector was integrated using HP 6890 ChemStation software. Individual FFA were identified and quantified using standards supplied by Sigma (Sigma Chemical Company, St. Louis, MO, USA).

Standard solutions with increasing concentrations of individual fatty acids and fixed concentrations of internal standards were used for the calculation of calibration curves.

2.5. Lipid oxidation

Hexanal was analysed using a solid phase micro-extraction (SPME) technique with a 75 μm Carboxen-PDMS fibre (Supelco, Bellefonte, PA, USA). A cheese sample (1.25 g) was placed in a 4 ml vial. The vial was placed for 5 min at 80 °C in a water bath with stirring to allow the sample to reach equilibrium and then the fibre was inserted into the vial through the septum. Fibre was exposed to the headspace above the sample for 30 min at 80 °C and desorbed in the injection port gas chromatography–mass spectrometry. Between runs, the fibre was cleaned for 10 min at 250 °C. Headspace hexanal was analysed using a gas chromatograph coupled to a mass selective detector 5973 (Hewlett Packard, Wilmington, DL, USA) and a CTC Analytics autosampler (Combipal, Agilent Technologies, Santa Clara, CA, USA). Analyses were performed using a Supelcovax (Supelco, Bellefonte, PA, USA) capillary column (60 m × 0.25 mm × 0.25 μm film thickness). SPME desorption was done at 280 °C for 5 min and the injector was set in splitless mode. Oven temperature was initially held at 40 °C for 10 min, increased to 110 °C at a rate of 5 °C min^{-1} followed by 10 °C min^{-1} to 240 °C, and finally held at 240 °C for 15 min. Helium was used as the carrier gas with a flow rate of 1 ml min^{-1}. Electron impact ionization was used at a voltage of 70 eV. Mass spectra of different treated samples were obtained with a scan range 35–250. Hexanal peak identification was performed by comparing the mass spectra with NIST08 and Wiley 7n1 libraries and by their retention time after comparison with a standard. Characteristic target ion was used to perform the semiquantitative determination of hexanal. Peaks areas (arbitrary units) were calculated from the total ion current.

2.6. Sensory evaluation

Flavour and aroma assessment of 60 day-old cheese samples was performed by a panel of 10 staff members experienced in the sensory assessment of cheeses. Gender distribution of the panel was even with a range from 30 to 55 years old. Panellists marked responses on a 9-point intensity scale where PA-cheeses were used as a control (0 = no differences with control, ±1 = minimal