J. Dairy Sci. 99:1–12
http://dx.doi.org/10.3168/jds.2015-10828

Volatile compound profiling of Turkish Divle Cave cheese during production and ripening

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ABSTRACT

The formation of volatile compounds in Turkish Divle Cave cheese produced in 3 different dairy farms was determined during production and ripening, revealing 110 compounds including acids, alcohols, ketones, esters, and terpenes. The presence and concentration of these volatile compounds varied between specific phases of the production and the 120-d ripening process. Smaller differences were also detected between cheeses produced at different farms. Carboxylic acids were established as a major class at the end of ripening. The relative amounts of acids and ketones increased until d 90 of ripening, whereas alcohols increased for the first 30 d and tailed off during the remaining part of the ripening process. The level of esters increased gradually until the end of ripening. Butanoic, acetic, and valeric acids, 2-butanol, 2-butanone, 2-heptanone, ethyl butanoate, α-pinene, and toluene were the most abundant compounds, likely contributing to the characteristic aroma of this traditional cheese.
Key words: volatile, ewe cheese, ripening, traditional, Divle

INTRODUCTION

Fermented milk products typically contain a wide range of flavors that are the result of biochemical conversions of milk components such as lactose, caseins, and milk fat. In cheese, the majority of flavors are derived from proteolytic processes and the subsequent conversion of AA (Visser, 1993). In industrial cheese productions, starter cultures are typically added to milk fermentations, greatly accelerating the process of flavor formation, as these bacteria harbor efficient proteolytic systems and AA-converting enzymes, resulting in the formation of a wide range of volatile compounds during production and ripening (Yvon and Rijnen, 2001; Smit et al., 2005).

Raw milk and raw milk cheeses typically also contain lactic acid bacteria and these strains possess AA-converting enzymes with higher or more diverse activities as compared with industrial dairy cultures, resulting in cheeses of more diverse and intense flavor (Centeno et al., 2002; Settanni and Moschetti, 2010). Moreover, other bacterial species, either naturally present in milk or contaminants added in during the handling of the milk, are present in raw milk, and albeit less controlled, play an important role in aroma development by their wide range of enzymes such as proteases and lipases (Chávez et al., 2010).

Divle Cave cheese is a semi-hard cheese made from raw semi-skimmed ewe milk. It is produced in May and June when milk is abundant, in Karaman, a rural region in the middle of Turkey. The ripening takes place in Divle Cave, which is located in the south of this region. The average temperature in the cave is 5 to 10°C with a humidity of 85 to 90%. The cheeses are produced without using any starter culture and are ripened for 4 mo in goatskin bags at a depth of approximately 70 m (Ozturkoglu-Budak et al., 2015). The surface of the goatskin bags changes from green to red during ripening in the cave, and this red color is the distinguishing characteristic of Divle Cave cheese (Figure 1), when compared with other spontaneously mold growing cheeses from Turkey (e.g., Kuflu cheese). Divle Cave cheese has a crumbly texture and a pungent flavor. Moreover, a large variety of aroma compounds occur in this cheese, likely as a consequence of the contribution of the diverse bacterial community present in raw milk (Ozturkoglu-Budak et al., 2015).

So far, studies on the flavor of Divle Cave cheese focused on the products at the end of ripening (Haya-

Received December 28, 2015.
Accepted March 9, 2016.
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loglu and Karabulut, 2013), but no studies have been published on the development of the volatile profile during production and ripening, which was the aim of this research. This paper presents data that contribute to a better characterization of Divle Cave cheese and these data will help to determine the authenticity of Divle Cave cheese in the future. The obtained data will also be useful for future comparative studies, both on mold-ripened cheeses and on raw ewe milk cheeses, enabling identifications of key flavor compounds that determine the unique taste of each of these cheeses.

MATERIALS AND METHODS

Cheese Production

Divle Cave cheeses were produced according to the traditional process and ripened in the Divle Cave (Ozturkoglu-Budak et al., 2015). Standardized semi-skimmed raw ewe milk was coagulated with calf rennet at 30 to 32°C for 80 min. The coagulum was cut into small sizes, heated to 55 to 60°C for 4 to 5 min, and the curd was placed into cotton cloths for whey drainage for 2 to 3 h. To remove the whey, pressure was applied to the cotton bag containing the curd at room temperature for one night. The resulting curd was rinsed with water for 24 h for complete removal of the whey. Subsequently, cheese blocks were dried by overnight storage at room temperature. The curd was broken into small pieces by hand, dry-salted, and tightly pressed into salted and dried goatskin bags. The goatskin bag was subsequently closed by sewing and pierced with needles to avoid serum loss. At the pre-ripening stage, goatskin bags were kept under cool storage conditions (15–20°C) until whey drainage stopped, in a process that took 7 to 10 d. Finally, the cheeses were left in Divle Cave for 4 mo for ripening.

Cheese Sampling

Cheese productions were carried out in 3 different dairy farms (**DF1**, **DF2**, and **DF3**) in the Karaman province of Turkey. Two independent batches were produced by each of the 3 farms with an interval of 15 d. We monitored and controlled all 6 productions and collected samples from each batch at the stages that the raw milk was obtained, and whey and curd were produced, and from cheeses at d 1, 30, 60, 90, and 120 during ripening. Each cheese sample consisted of one goatskin bag of cheese (3–5 kg). Ten goatskin bags of cheese were prepared per each batch and put in the cave. At each sampling day, duplicate goatskin bags were collected from the cave for analyses. A total of 60 different goatskin bags were analyzed in both batches produced in 3 DF. All samples were transported to the laboratory at 4°C, and divided into different aliquots that were stored at −20°C before analysis. Analyses were performed in triplicate from different parts of cheeses in each goatskin bag. The reported values are the means of the 2 independent batches per DF.

Physicochemical Analyses

Titratable acidity (as % of lactic acid) was measured according to AOAC International (1995). The pH was measured by direct insertion of a pH-meter electrode (Mettler Toledo MP 225, Columbus, OH) into the slurry sample obtained by maceration of a 10-g sample with 10 mL of distilled water. Dry matter was analyzed by the oven drying method at 102°C (IDF, 1982), whereas salt content was determined by titration with $AgNO_3$ (IDF, 1988). Fat content was measured by the Gerber-Van Gulik method (Ardo and Polychroniadou, 1999), and water-soluble nitrogen (**WSN**) as well as total nitrogen (**TN**) were determined by applying the micro-Kjeldahl method on fractions of the cheese prepared as described by IDF (1993). Ripening index (RI) values were calculated according to the ratio of WSN content to total nitrogen content [RI = (WSN/TN) × 100]. In the milk, whey and curd samples the pH, titration acidity, DM, and fat content was measured, whereas in the cheese samples these measurements were complemented with WSN and TN measurements. All analyses were performed in triplicate.

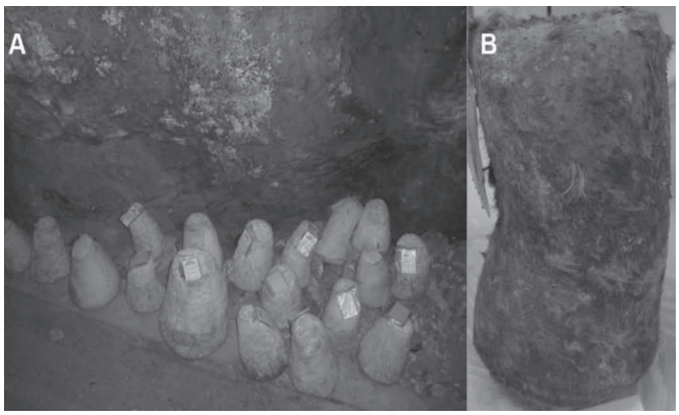

Figure 1. Growth of microorganisms on the surface of goatskin bags during the ripening period of Divle Cave cheese. (A) A view from the cave on d 30 during ripening, (B) appearance of the goatskin bag at the end of the ripening (on d 120). This figure is reprinted by permission of Int. Dairy J. (Ozturkoglu-Budak et al., 2015; doi: 10.1016/j.idairyj.2015.09.011). Color version available online.