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ORIGINAL PAPER

Volatile profile evolution and sensory evaluation of traditional skinbag Tulum cheeses manufactured in Karaman mountainous region of Turkey during ripening

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Abstract

Skinbag Tulum cheeses produced by traditional methods in the Karaman region of Turkey were selected as research material. The volatile fraction was determined during 180 days of ripening by SPME–GC–MS technique. In the meantime, sensory evaluation was performed on the 90th and 180th days of the ripening. According to the results of volatile component analysis, 12 carboxylic acids, 9 alcohols, 16 esters, 10 ketones, 2 aldehydes, 4 terpenes, and 3 hydrocarbons were detected. Among them, the dominant groups were carboxylic acid, esters, ketone, and alcohols, respectively. Octanoic and hexanoic acids have been found to be the most typical carboxylic acids in Tulum cheeses. The predominant esters were ethyl acetate and methyl decanoate. Some other notable volatile components were 2-butanone, isoamyl alcohol, and benzaldehyde. For all sensorial characteristics except odor, ripening time was statistically significant and the highest scores were obtained on the 90th day of the ripening.

Keywords Volatile compounds · Tulum cheese · Solid-phase microextraction and gas chromatography–mass spectrometry (SPME–GC–MS) · Sensory evaluation

Introduction

Tulum cheese is typically manufactured in Turkey, and its main characteristics are white and cream-colored, high dry matter and fat contents, not easily dispersed semi-hard, homogeneous texture, and acidic taste caused by long-term ripening [1]. More than 50 varieties of cheese are present in Turkey and Tulum cheese is the most produced one after Beyaz and Kasar cheeses [2]. Furthermore, consumer demand for Tulum cheese has increased over the years [3]. Since a standard technique is not applied in the production of Tulum cheese in our country, it is generally produced by traditional methods in family-owned companies and primitive dairy farms between March and July months. Goat and sheep milk is traditionally preferred in the production of Tulum cheeses; however, cow milk is also used recently due to the increasing demand for Tulum cheese consumption. Although traditional manufacturing methods are maintained for Tulum cheese, various new technological advantages have been incorporated into artisanal production over time [2].

Traditional Tulum cheese is generally referred to different names depending upon the region where it is produced in Turkey. Şavak or Erzincan which is known as the most popular Tulum cheese produced in the Erzincan region. Likewise, Çimi Tulum (Antalya), Afyon Tulum (Afyon), Kargi Tulum (Çankırı, Çorum), Isparta Tulum (Isparta) and İzmir (brine) Tulum cheeses are also known and frequently consumed cheeses [4]. Tulum cheese produced in Divle Obruk cave in Karaman region has also received a protected designation of origin (PDO) in 2017 with the name of Divle Obruğu Tulum cheese [5]. Due to the climatic changes in the regions where cheese is produced, the differences in the type and

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composition of the milk lead to the presence of different qualities of Tulum cheese in the market [6].

Cheese flavor is one of the most important parameters for both consumer preferences and the quality standards of the manufacturer [7]. The flavor, aroma, and quality of cheese are determined by the biochemical and chemical reactions such as glycolysis, proteolysis, and lipolysis which occur during ripening and by volatile and non-volatile compounds originating from the fat, protein, and carbohydrate catabolism [7, 8]. Non-volatile compounds affect the taste of cheeses, while volatile compounds contribute to both taste and odor [9]. More than 600 volatile compounds have been identified in cheese, but only a small amount of them can be considered as "flavor-impact" volatile compounds [10]. Furthermore, each cheese has a different volatile composition that provides its own flavor. Generally, the main groups of volatile compounds groups which are noteworthy for cheese flavoring are esters, ketones, alcohols, carboxylic acids, sulfur compounds, and terpenes [11]. Most of the volatile aroma components come from proteolytic and lipolytic activities and conversion reactions of amino acids. Since amino acid catalyzing enzymes found in cheese cause the formation of a wide variety of volatile components during production and ripening, they greatly accelerate and affect the flavor formation process [8]. These reactions and volatile profiles are affected by parameters such as origin of the milk, milk treatment method, microbial activity, and ripening conditions [12].

Many methods are available for the study of volatile compounds. Mostly, GC (gas chromatography)–MS (mass chromatography) are utilized for volatile compounds analysis [13, 14]. These methods usually include extraction and pre-concentration of the volatile fraction as the primary step. In these difficult processes, researchers have been forced to search for new, easier and faster methods such as solid-phase micro-extraction (SPME). The SPME technique has been frequently used for the concentration of volatile and non-volatile compounds [15, 16] and also there are some works on several types of Tulum cheeses using the SPME technique [4, 11, 17–20]. From these studies, it has been seen that the differences in milk composition depending on the geographical location and milk types, in traditional production methods, and in ripening time affect the volatile components in cheeses.

This study focused on the identification of the volatile fractions using the SPME–GC–MS technique in Tulum cheeses produced by traditional methods using various types of milk (goat, sheep, and cow) in the Karaman region. For this study, eight samples of Tulum cheese were taken from different producers. The manufacturing of the Tulum cheese samples was performed by the nomads in the highland villages (Habipler, Taşkale, Berendi, and Büyükkarapınar villages) of Karaman region where the elevation from sea level is approximately 1400–1650 m and the temperature difference between day and night is very high. To our knowledge, this is the first study in which the dominant volatile components of the skin bag Tulum cheeses locally produced in the Karaman mountainous region of Turkey were analyzed over the 180 days of ripening in detail. Moreover, changes in basic sensory properties during the ripening period for Tulum cheeses were investigated and these characteristics were examined in relation to the changes in the volatile fraction during ripening.

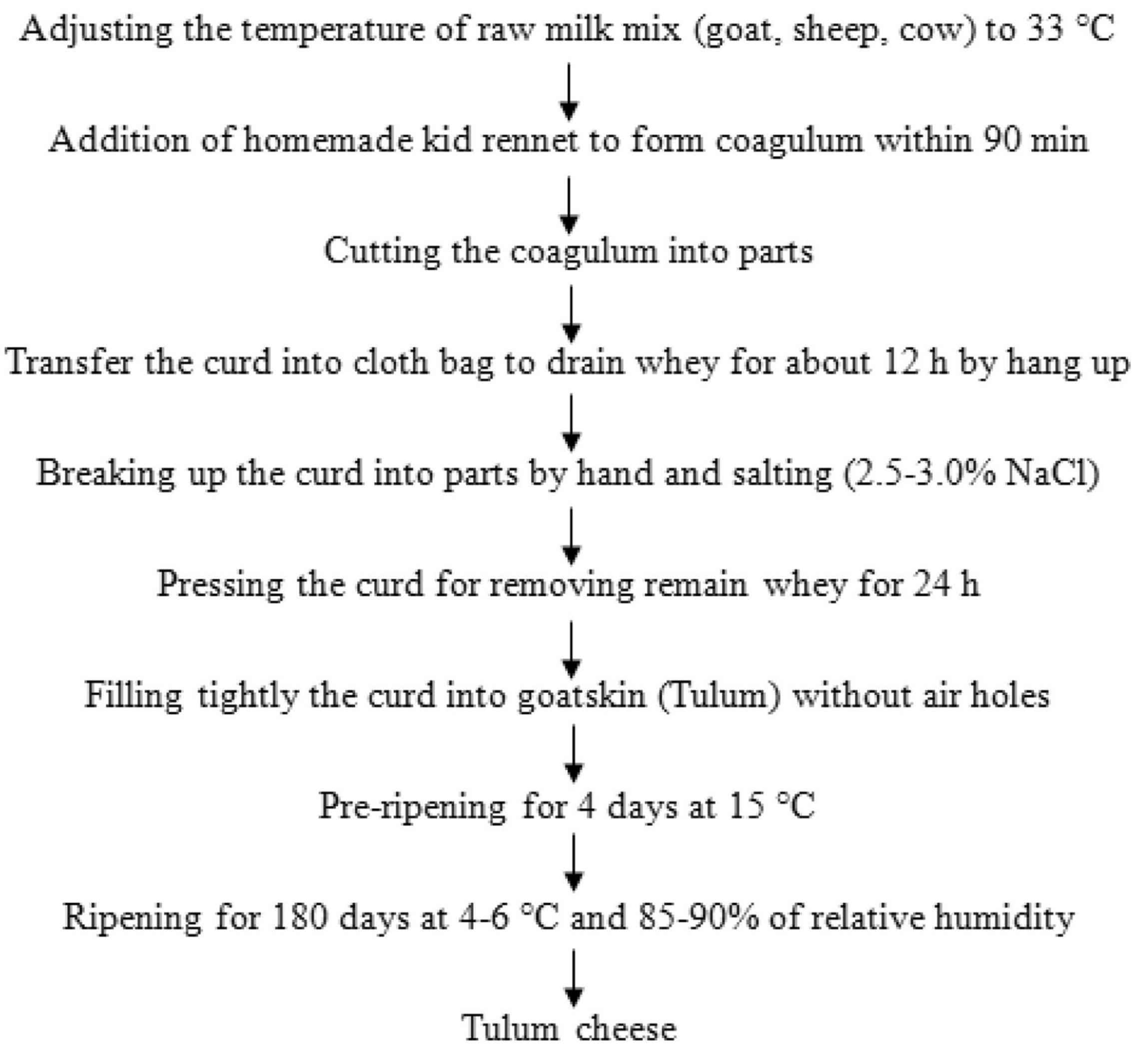
Materials and methods

Tulum cheeses

Tulum cheeses were manufactured by traditional methods with 8 different local producers (8 biological repetitions) in Karaman region (Turkey), mostly using raw goat's (60–70%), sheep's (20–30%), and a small amount of cow's (10%) milk. The production method of Tulum cheese is presented in Fig. 1. Tulum cheeses were supplied in 2.5–3.0 kg weight. Unripened Tulum cheeses were delivered to the laboratory in the cold chain and ripened under similar conditions to those used by traditional producers. Tulum cheeses pre-ripened for 7 days at 15 °C and then were ripened for 180 days at 4 °C with relative humidity of 85%. Tulum cheeses were sampled at 7, 15, 30, 60, 90, and 180 days of ripening. After removing the skin bag, whole cheese mass was mixed homogenously, and then experimental samples were taken. A total of 48 (8*6) cheeses was analyzed along the ripening periods.

Volatile composition by SPME–GC–MS

The volatile composition of the Tulum cheeses was determined using the solid-phase microextraction (SPME) technique using an Agilent 7890 GC system (Agilent Technologies, Santa Clara, CA) coupled to a mass spectrometer (Agilent 5975C VL MSD with Triple-Axis detector) (GC–MS) [18, 21]. A 3.0 g portion for each cheese sample was placed in a 15 mL vial, followed by 10 µL of internal standard containing 81 ppm of 2- methyl-3-heptanone, 2-methyl-pentanoic acid (Sigma–Aldrich Co., USA) in methanol for determining the relative amount of volatile compounds. The prepared sample was kept at 50 °C for 30 min to equilibrate volatile compounds in the headspace. Extraction of volatiles was carried out using a solventless extraction technique. Essentially, extraction was achieved by inserting a 75 µm carboxen polydimethylsiloxane (CAR/PDMS) fiber (Supelco, Bellefonte, PA, USA) into the vial and exposing it to the headspace for 30 min at 50 ºC. The fiber was then left into the injection port of the GC for 3 min



Adjusting the temperature of raw milk mix (goat, sheep, cow) to 33 °C
Addition of homemade kid rennet to form coagulum within 90 min
Cutting the coagulum into parts
Transfer the curd into cloth bag to drain whey for about 12 h by hang up
Breaking up the curd into parts by hand and salting (2.5-3.0% NaCl)
Pressing the curd for removing remain whey for 24 h
Filling tightly the curd into goatskin (Tulum) without air holes
Pre-ripening for 4 days at 15 °C
Ripening for 180 days at 4-6 °C and 85-90% of relative humidity
Tulum cheese

Fig. 1 The traditional production method of Tulum cheese

at a temperature of 250 ºC, with helium as the carrier gas at a flow rate of 1.0 mL/min. The volatile compounds were separated on a DB-Wax column (30 m × 0.25 mm × 0.25 µm; J&W Scientific, Folsom, CA, USA). The initial condition of the program was performed at 40 °C for 10 min, followed by elevation of the temperature to 110 °C at a rate of 5 °C/min and 240 °C at a rate of 10 °C/min with a final extension of 5 min at 250 °C. Mass spectra were recovered in the electron impact mode at an ionization voltage of 70 eV and data were collected at a rate of 3.2 scans/s over a range of *m/z* 35–500. Identification of volatile components was achieved by comparison of their mass spectra with those in the libraries of National Institute of Standards and Technology (NIST) and Flavor (Agilent MSD Chemstation, Santa Clara, CA). Relative amounts of volatile components were calculated by proportioning the peak area ratio of volatile components and peak area ratio of the internal standard, relative to the concentration of the internal standard. Cheese samples were analyzed in duplicate. The averages of the amounts of volatile components are given as mg/kg.

Sensory analysis

The sensory analysis was carried out by 25 semi-trained panelists, who had no experience but took part in previous training sessions in Selcuk University Food Engineering Department, where characteristic definitions were clarified. During training sections, commercial Tulum cheese was used to show and explain the characteristic properties of Tulum cheese for panelists. The analysis was done in 2 repetitions. Samples were coded differently in each repetition. Sensory evaluation of cheese samples was carried out according to the instructions of the Turkish Standard (TS) 3001 containing "100 Full Point Evaluation System" [22]. According to this standard “appearance”, "body and texture", "odor", and "flavor" characteristics of Tulum cheeses were evaluated over 30, 20, 10, and 40 points, respectively. Sensory analysis of the samples was done at 90 and 180 days of ripening, due to possible pathogen risks if consumed before (legislation does not allow the consumption of these cheeses before 90 days of ripening since they are made with raw milk) [22].

Statistical analysis

The obtained data from sensorial analysis were subjected to two-sample independent *t* test to compare the means of each sensorial attribute with a confidence interval set at 95%. All the data were expressed as a mean ± standard deviation of the duplicates.

Results and discussion

Volatile fraction

According to the results of volatile compounds analysis by SPME–GC–MS technique, a total of 56 different volatile compounds including different 12 carboxylic acids, 16 esters, 10 ketones, 9 alcohols, 2 aldehydes, 4 terpenes, and 3 hydrocarbons were detected throughout all periods. The distribution of all major component groups during each ripening period is presented in Fig. 2. Besides, all volatile compounds detected in cheeses are shown in Table 1 in detail.

Carboxylic acids

Acids are especially important components in the aroma of ripened cheeses. Most of the acids detected in cheeses generally occur as a result of lipolysis of milk fat [23]. Otherwise, carboxylic acids are regarded as important precursor molecules for the formation of substances such as secondary alcohols, methyl ketones, esters, and lactones that contribute to the aroma [24]. Fatty acids with long-chain carbon atoms (C14–C18:3) can be formed by lipolysis of triglycerides, those with medium-chain carbon atoms (C10–C12) can be formed by degradation of lactose and amino acid, and short-chain fatty acids can also be formed by the oxidation of aldehydes, ketones and esters [25]. On the other hand, Vélez et al. [26] have reported that short and medium-chain fatty acids contribute significantly to the taste of various cheeses. In this study, carboxylic acids have been the predominant group of volatile compounds and those acids constantly observed during the entire ripening period were; octanoic acid, heptanoic acid, acetic acid, butanoic acid, 3-methyl isovaleric acid, 2-methyl valeric acid, 2-methyl pentanoic acid, 2-methyl hexanoic acid, and decanoic acid. On the other side, acids such as pentanoic, undecanoic, lauric, and 2-methyl butanoic were detected only in certain periods. Similar to our study, Delgado et al. [27], Ozturkoglu-Budak et al. [28], Pino et al. [29] and Kondyli et al. [30] emphasized carboxylic acids as the most important flavor component in cheeses.

Acetic acid was found as the most dominant acid at the beginning of the ripening, while octanoic acid, pentanoic, acid and hexanoic acid were detected at the end of the ripening. Hexanoic acid and acetic acid reached their highest values on the 60th day of the ripening period. The amount of acetic acid showed fluctuation throughout the ripening period and it was seen at the highest amount on the 90th day of ripening. On the other hand, butanoic and decanoic acids were carboxylic acids that tended to increase continuously during ripening.

On the whole, the most common carboxylic acids were octanoic acid, hexanoic acid, acetic acid, valeric acid, and butanoic acid, respectively, as shown in Fig. 3. Hayaloglu et al. [2] found that the amount of butanoic acid detected as one of the principal acid components in cheeses matured in skin bag compared to that in plastic containers. Likewise, Yilmaz et al. [31] found that the dominant free fatty acid is butanoic acid in Tulum cheeses. Besides, Qian and Burbank [32] reported that the octanoic acid, which we encountered in the highest amount, was detected more in goat milk, and Ercan et al. [33] stated that this acid has goatish, cheesy, and waxy aroma notes specific to cheeses. Hexanoic acid is also known to be one of the acids that give cheese its unique aroma, especially in cheeses made from goat's milk. Consistent with our results, Pino et al. [29] working on Nicastre cheese produced from raw goat milk asserted that mostly the group of fatty acids was found as volatile compounds in cheese and among them, as octanoic acid and hexanoic acid were more abundant. Although, Hayaloglu et al. [2], Cakir et al. [4] and, Akarca [19] found butanoic acid as the highest acid component in Tulum cheeses produced using sheep's milk, in our study goat's, sheep's, and cow's milk were used in certain proportions, so the butanoic acid was not seen as the most dominant acid as in the studies mentioned. Also, the findings in this study on hexanoic and octanoic acid amounts are not similar to those reported by Ceruti et al. [34] who stated that no hexanoic and octanoic acids were detected for Reggianito cheeses that is a hard cheese such as Tulum cheese. Butanoic acid, which tends to increase continuously until the end of ripening, has been reported to give a rancid-cheese-like aroma [17] whilst acetic acid gives the pungent and vinegar aroma to cheese such as Feta and Domiati [27]. Our results on acetic acid are corroborated by the findings of Hayaloglu et al. [17] and Gursoy et al. [35] who reported that acetic acid was the highest carboxylic acids in Gökceada Tulum cheese and Sögle Tulum cheese. Likewise, Ozturkoglu-Budak et al. [28] stated that

Table 1 Distribution and classification of volatile compounds during the ripening of traditional skinbag Tulum cheese

CAS no.	Volatile compounds	Ripening days					
		7	15	30	60	90	180
	Carboxylic acids[1]						
64–19-7	Acetic acid	13.48	15.72	14.31	36.36	42.1	18.46
107-92–6	Butanoic acid (butyric acid)	3.16	8.24	10.69	16.15	20.5	29.6
503–74-2	Isovaleric acid (3-methyl-butanoic acid)	0.12	16.5	0.5	5.54	33.34	0.82
109–52-4	Valeric acid (pentanoic acid)	0.4	ND	13.04	41.64	13.83	43.41
116–53-0	2-Methyl-butanoic acid	0.07	1.36	0	1.44	4.82	0.3
142–62-1	Hexanoic acid (caproic acid)	11.62	18.79	18.08	66.23	43.27	37.17
97–61-0	2-Methyl pentanoic acid	5.82	5.81	8.68	8.66	5.31	10.09
4536–23-6	2-Methylhexanoic acid	0.82	1.79	0.88	1.04	2.78	1
124–07-2	Octanoic acid (caprylic acid)	4.56	8.03	12.71	56.92	52.08	82.78
334–48-5	Decanoic acid (capric acid)	1.5	2.7	4.13	13.68	19.1	21.28
112–37-8	Undecanoic acid	0.25	0.62	ND	11.73	0.2	ND
143–07-7	Lauric acid (dodecanoic acid)	ND	ND	ND	ND	0.05	6.97
	Total concentration	41.8	79.56	83.02	259.39	237.38	251.88
	Esters[1]						
141–78-6	Ethyl acetate	67.69	20.6	41.98	14.06	13.63	1.58
79–20-9	Methyl acetate	ND	19.16	ND	ND	ND	ND
105–54-4	Ethyl buthyrate	0.03	ND	1.7	0.04	2.08	0.64
623–42-7	Methyl buthyrate	ND	ND	4.41	7.49	0.9	1.33
106–70-7	Methyl hexanoate (Methyl caproate)	0.53	6.11	3.54	8.51	6.58	25.81
123–92-2	Isoamyl acetate	7.94	24.51	7.17	15.25	2.54	ND
106–30-9	Ethyl heptanoate	0.06	0.25	0.12	2.45	0.25	4.64
106–27-4	Isoamyl butyrate	ND	0.82	0.04	0.15	0.14	0.14
111–11-5	Methyl octanoate (octanoic acid methyl ester)	0.21	5.47	0.24	18.91	12.96	42.53
106–32-1	Ethyl octanoate (octanoic acid ethyl ester)	1.56	2.85	6.06	5.13	18.11	8.47
103–45-7	Phenethyl acetate	3.11	20.94	2.73	33.42	5.96	3.84
110–42-9	Methyl decanoate	0.23	0.2	30.05	20.34	15.01	51.04
110–38-3	Ethyl decanoate	ND	0.26	1.37	4.62	25.43	3.91
123–9-5	Ethyl nonanoate	ND	1.68	ND	2.57	1.15	2.68
106–33-2	Ethyl laurate	ND	0.07	0.04	1.16	0.32	0.46
111–82-0	Methyl laurate	ND	2.91	4.38	ND	ND	ND
	Total concentration	81.36	105.83	103.83	134.1	105.06	147.07
	Ketones[1]						
67–64-1	Aceton (2-propanone)	ND	ND	28.07	ND	0.19	0.25
513–86-0	Acetoin (3-hydroxy-2-butanone)	5.32	5.12	1.68	1.81	4.97	4.66
78–93-3	2-Butanone	0.04	18.53	12.53	30.04	57.86	80
107–87-9	2-Pentanone (pentanone)	0.13	0.18	21.84	0.13	2.29	21.73
13706–86-0	5-Methylhexane-2.3-dione	ND	ND	1.49	2.09	ND	0.44
110–43-0	2-Heptanone	ND	ND	ND	ND	1.97	1.84
111–13-7	2-Octanone	0.01	ND	ND	1.05	0.19	0.63
821–55-6	2-Nonanone	ND	3.9	97.01	0.85	6.32	63.94
5009–32-5	8-Nonen-2-on	ND	ND	2.7	0.04	1.54	3.42
112–12-9	2-Undecanone	0.01	0.02	6.26	0.05	1.85	2.79
	Total concentration	5.51	27.75	171.58	36.06	77.18	179.7
	Alcohols[1]						
64–17-5	Ethanol (ethyl alcohol)	3.69	4.33	4.04	3.08	11.93	0.89
513–85-9	2.3-Buthanediol	2.75	2.89	4.11	6.87	3.73	0.94
78–92-2	2-Buthanol	0.02	3.5	5.13	15.96	25.09	12.53

Table 1 (continued)

CAS no.	Volatile compounds	Ripening days					
		7	15	30	60	90	180
123–51-3	Isoamyl alcohol	10.79	18.74	10.03	17.51	11.42	3.28
6032–29-7	2-Penthanol	ND	ND	5.31	0.02	3.69	1.58
543–49-7	2-Heptanol	0.02	1.27	9.14	3.79	6.21	18.35
60–12-8	Phenylethyl alcohol	1.84	1.67	1.28	1.66	3.64	1.86
143–08-8	1-Nonanol	0.23	0.33	0.21	0.12	0.12	0.14
628–99-9	2-Nonanol	ND	0.01	ND	0.19	0.26	1.58
	Total concentration	19.34	32.74	39.25	49.2	66.09	41.15
	Aldehydes[1]						
2100–17-6	4-Penthanal	0.21	0.09	0.31	0.3	0.48	ND
100–52-7	Benzaldehyde	0.08	0.15	0.09	3.21	1.86	3.08
	Total concentration	0.29	0.24	0.4	3.51	2.34	3.08
	Terpenes[1]						
80–56-8	Apha-pinene	2.29	1.77	4.57	2.51	1.88	2.48
5989–27-5	D-Limonene	0.06	0.07	0.05	0.07	0.05	0.13
99–85-4	Gamma-terpinene	0.82	0.52	0.47	1.34	ND	1.47
87–44-5	Beta-caryophyllene	0.01	0.01	0.03	0.03	0.08	0.07
	Total concentration	3.18	2.37	5.12	3.95	2.01	4.15
	Hydrocarbons[1]						
108–88-3	Toluene	1.05	0.9	1.41	1.26	1.09	2.27
100–42-5	Styrene	2.8	7.09	5.83	12.45	8.43	6.68
99–87-6	p-Cymene	0.58	0.54	0.5	0.74	0.75	0.87
	Total concentration	4.43	8.53	7.74	14.45	10.27	9.82

Average values are given in mg/kg

ND Not detected

[1] The values in the table are the average amounts of volatile components ($n = 8$)

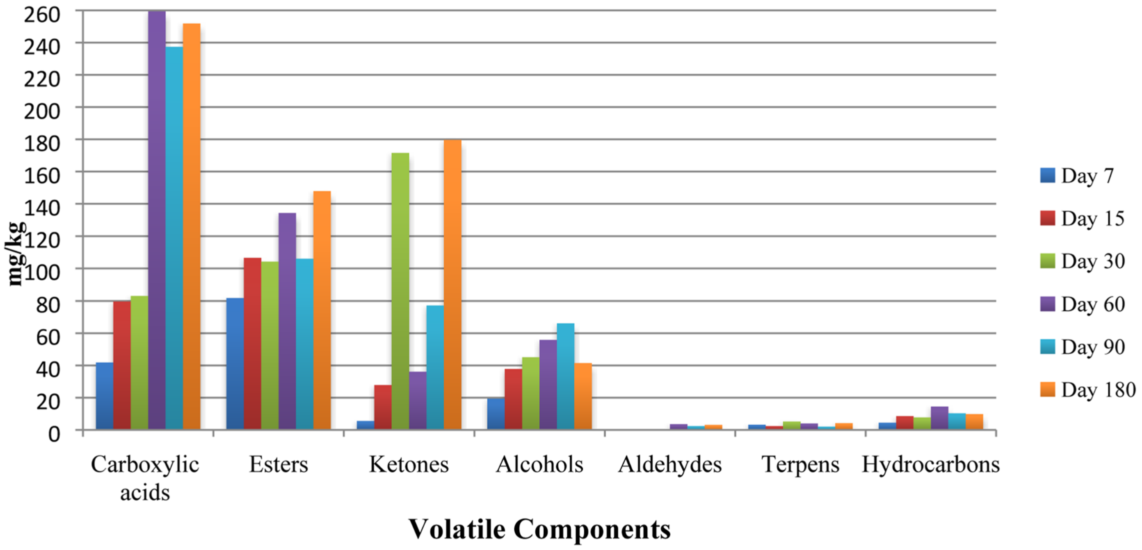


Fig. 2 Distribution of total concentrations of volatile component groups during each ripening periods ($n = 8$)

acetic acid was identified as the most detected carboxylic acid amongst all in Divle Tulum cheese types.

Alcohols

In general, alcohols in cheeses are formed by metabolic pathways such as lactose metabolism, methyl ketone reduction, amino acid metabolism, linoleic and linolenic acid degradation. Furthermore, secondary alcohols are formed by enzymatic reduction of the respective methyl ketones, while branched-chain primary alcohols are formed by reduction of aldehyde derived from leucine [27]. Alcohols were asserted as one of the main group of volatile compounds in semi-hard and long-aged cheeses such as Tulum cheese, Cheddar cheese and raw sheep milk cheeses [10, 36]. Ethanol, which can be formed by the degradation of lactose by the hetero-fermentative lactic acid bacteria and by the catabolism of alanine amino acid, is an important precursor in the formation of esters [8, 11]. Although ethanol a limited aromatic role in cheeses, the progressive ripening time of cheese can promote the rapid conversion of ketones and aldehydes to their primary and secondary alcohols [37]. At the same time, ethanol was notified to be the main alcohol in many goat's milk cheeses [38].

Considering the alcohol content of the cheeses of the Karaman region, a total of 9 different alcohols were identified as seen in Table 1. At the initial days of ripening, isoamyl alcohol, ethanol and 2,3-butanediol were the alcohols found at the highest levels, whereas 2-heptanol, 2-butanol and isoamyl alcohol became dominant at the end of the ripening (Fig. 4). Ethanol and 2–3-butanediol were observed over the whole ripening time but they remained in extremely low amounts at the end of the ripening. Isoamyl alcohol, which could be related to the reduction in the aldehyde produced from leucine by Strecker degradation [27], took place as the predominant alcohol until day 60, but it gradually decreased until day 180. Besides, 2-butanol reached its higher level and became the predominant alcohol on day 90 and then decreased. The reduction in alcohol amounts is probably due to the reaction of alcohol and acid that occur to form esters [27]. 2-heptanol reached the highest amount on the 180th day, and it was detected in low amounts in previous days. Phenylethyl alcohol was also found in all periods of ripening, and its highest level was detected on the 90th day (Fig. 2).

When it comes to total alcohol amount, the most common alcohols were isoamyl alcohol, 2-butanol, and 2-heptanol. Our findings are supported by the study of Ocak et al. [39] who produced cheese by mixing cow, ewe and goat milks in different proportions and stated that the most dominant alcohol in the volatile component of these cheeses was isoamyl alcohol. Similarly, Ozturkoglu-Budak et al. [28] identified isoamyl alcohol as the dominant alcohol in Divle Tulum cheeses. Curioni and Bosset [10] found that 2-butanol is the predominant alcohol in their study on Tulum cheese produced from raw sheep's milk. 2-butanol has been reported to provide a fruity aroma note. 2-butanol and 2-heptanol have been detected as the main odor component in cheeses such as Gorgonzola and Grana Padano with the gas chromatography–olfactometry method [10]. Avşar et al. [40] found that 2-heptanol was the most abundant alcohol in Tulum cheeses produced by mixing goat,

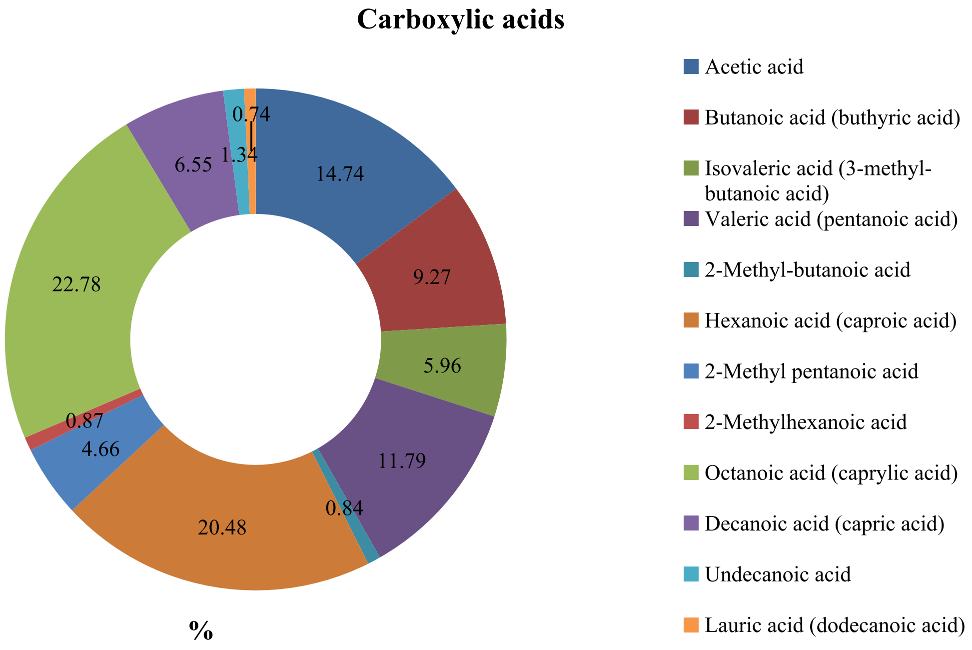
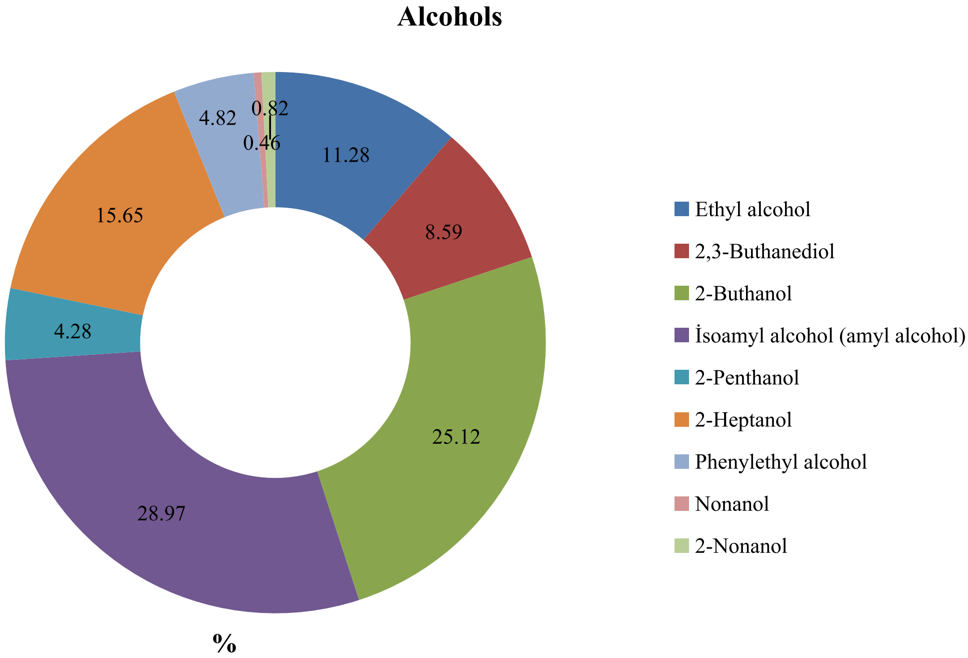


Fig. 3 Average distribution (%) of carboxylic acid detected over all ripening periods in traditional skin bag Tulum cheeses (*n* = 8)

Fig. 4 Average distribution (%) of alcohols detected over all ripening periods in traditional skin bag Tulum cheeses ($n=8$)



sheep, and cow milk in the Middle Taurus. On the other hand, 2-heptanol is characterized with fruit, soil, grass and sweet aroma notes, and 2-pentanol provides grass, fruity and fresh aroma notes, 2-nonanol presents oily and grassy aroma notes [10].

Esters

In Tulum cheeses, carboxylic acids were found the highest in terms of quantity, while esters were found the most diverse group. The biosynthesis of esters is carried out via enzymatic or chemical reactions of short- and medium-chain fatty acids with alcohols. These formation reactions in cheeses may either occur spontaneously or mediated by the esterase activities of lactic acid bacteria [41]. Esters have floral and fruity flavor notes that help minimizing the bitterness of amines and the sharpness of fatty acids and particularly ethyl esters are known for their important role in the formation of a fruity aroma note in cheeses [38, 42]. Esters are known to generally have low perception threshold values [42]. Therefore, they give a very important contribution to cheese aroma.

When the cheeses produced in the Karaman region were examined in terms of their ester content, ethyl acetate was found in very high amounts at the beginning of the ripening, but it decreased gradually and remained at a low level until the end of the ripening. Likewise, it was determined that isoamyl acetate was high in the initial days of ripening, but it was not found in cheese at the end of maturation. Hayaloglu et al. [2] stated that they identified 16 esters in the 90th ripening day of Tulum cheeses and reported that the most abundant ester was ethyl acetate. It was observed that phenethyl acetate was also high at the beginning of the ripening. The esters remaining at the highest rate at the end of storage were determined as methyl decanoate, octanoic acid methyl ester (methyl octanoate), and hexanoic acid methyl ester (methyl caproate), respectively. Ethyl octanoate was also one of the highly determined esters in cheese samples. Similar to our study, Hayaloglu et al. [17] reported that 20 different esters in Gokceada cheese which are traditionally produced from goat milk, and that most of them were ethyl esters. The researchers also stated that many traditional kinds of cheeses manufactured from goat milk contained ethyl ester. Medjoudj et al. [37] reported that ethyl octanoate, ethyl decanoate, and ethyl hexanoate were found as the most dominant esters in Algerian traditional cheese called Bouhezza. Also, Liu et al. [43] reported that ethyl octanoate is responsible for pear, pineapple, apricot, and flower aroma notes whilst ethyl acetate provides solvent, pineapple flavor notes, and ethyl hexanoate gives banana, pineapple, wine flavor notes. According to our results, methyl decanoate, octanoic acid methyl ester and hexanoic acid methyl ester were in small amounts at the beginning of the ripening, and reached their highest levels at the end of the ripening. Liu et al. [43] reported that esterification events were supported by environments with low water activity. In this case, the increase in the concentrations of the esters mentioned can be due to decreasing water activity of the Tulum cheeses with progressive ripening days.

Ketones

Ketones are formed by the enzymatic oxidation of fatty acids to β-keto acids and then their decarboxylation to methyl ketones [8]. Methyl ketones most commonly found in cheeses such as 2-nonanone, 2-heptanone, which are known to be responsible for fruity, moldy, sweet, rose or tea-like aromas, and blue-cheese notes, are widely detected especially in Italian cheeses which is in accordance with our results [10]. Furthermore, it has been reported that ketones and especially methyl ketones are the main ingredients contributing to the aroma of moldy cheeses [8].

Regarding ketone composition of Tulum cheeses in our work, acetoin (3-hydroxy-2-butanone) was found in high amounts on the first day of ripening. Overall time of ripening, the most abundant ketones were 2-butanone, 2-nonanone, 2-pentanone, and acetoin. It was observed that 2-butanone, which could not be detected at the beginning of ripening, began to be detected later and step by step increased during ripening. It contributed significantly to the aroma by being the most abundant ketone at the end of ripening. It was known that 2-butanone, which are produced by carbohydrate metabolism, gives buttery and sour milk aroma in many cheeses [8]. Similarly, Hayaloglu et al. [2] found that the highest methyl ketone detected in Tulum cheeses ripened into skin bag material was 2-butanone. Although 2-nonanone had a fluctuating course, it was seen in high amounts at the end of ripening. In this present study, 2-pentanone was detected in high amounts especially at the end of the 30th day and at the end of ripening. Other ketones were observed in incredibly low amounts during ripening. Our findings are in accordance with earlier results by Ozturkoglu-Budak et al. [28] who found 2-butanone and 2-pentanone to be the dominant ketones in Divle Tulum cheese, and by Barron et al. [44] observed that the predominant methyl ketones of sheep's milk were 2-butanone and 2-pentanone with dynamic headspace method. Similarly, Bontinis et al. [38] reported that 2-butanone was the dominant ketone in goat milk cheeses, whilst Pino et al. [29] found 2-nonanone as the dominant ketone in traditional Nicastrese cheese made from goat's milk in parallel with our observations. Hayaloglu et al. [2] stated that the concentration of methyl ketones, which are reduced to their corresponding secondary alcohols, characterizes the aroma profile of Tulum cheese.

Aldehydes

Aldehydes from the autoxidation of carboxylic acids have a role in the flavor development of cheese, despite their low odor thresholds and quick reduction to alcohols and acids, but their role in flavor is not as important as other lipid metabolites [45, 46]. Aromatic chain aldehydes are strong odor volatiles. Therefore, its effect on the cheese flavor is strong in ripened cheeses [47]. Most aldehydes give the cheese a grass, bitter almond, herb and malt flavors [10]. Detection of low amounts of aldehyde in cheeses shows that optimal ripening conditions are optimal and that aroma is appropriate, and the presence of excess aldehyde may be a sign of aroma disturbances [41]. Only two aldehydes; benzaldehyde and 4-pentanal were detected in Tulum cheeses. 4-pentanal and benzaldehyde were observed in the highest amounts on the 90th and 180th days, respectively. 4-pentanal was not detected as it was completely decomposed on the end of storage. Benzaldehyde is an aromatic aldehyde that gives the cheese bitter and almond flavor [48]. Hayaloglu and Karabulut [11] detected benzaldehyde in goat milk cheeses at similar levels. Aminifar et al. [41] detected small amounts of aldehyde in the Ligvan cheese as in our study, associated these levels with optimal cheese aging. What is more, Moio and Addeo [49] stated that high amounts of aldehyde may cause unwanted aromas in their study with the vacuum distillation extraction method on Grana Padano cheeses.

Terpenes

Terpenes are volatile constituents associated with animal feeds, pastures, green forages and plants that make up the forage mixes of pastures in traditional cheeses produced and sold in meadows and especially high mountains [50, 51]. Some researchers stated that the contribution of terpenes to the formation of cheese aroma is not clear, but these components are frequently identified in cheeses produced from goat and sheep milk [38, 52]. In this current study, a total of 4 terpenes were detected. Sorting according to the quantity of terpenes obtained were α-pinene, γ-terpinene, limonene, and β-caryophyllene, respectively. The most abundant compound at the beginning and end of the ripening was α-pinene. It is known that α-pinene is one of the most abundant terpene components in milk [53]. Also, this is corroborated by data of Ozturkoglu-Budak et al. [28] who studied Divle Tulum cheeses. The presence of α-pinene, β-caryophyllene could be attributed to the native feed of animals in the Karaman region. At the same time, terpenes such as m-cymene and β-caryophyllene may be "marker" of Karaman pastures which sheep graze, as claimed by Cornu et al. [53]. Kondyli et al. [30] also claimed that α-pinene was the most concentrated terpene component in cheeses produced from goat's milk. Limonene, on the other hand, is a volatile ingredient with a citrus aroma. In a previous study conducted by Medjoudj et al. [37], who studied the volatile ingredient in traditional Algerian cheese known as Bouhezza, limonene, α-pinene, and γ-terpinene were determined as the most abundant terpenes in cheeses at the end of ripening.

Table 2 Mean results of sensorial attributes of traditional skin bag Tulum cheeses on the 90th and 180th days of ripening

Ripening days	Sensorial attributes (points)				Total score (100)
	Appearance (30)[1]	Body and texture (20)[2]	Odor (10)[3]	Flavor (40)[4]	
90	24.42 ± 2.57a	16.57 ± 2.44a	8.14 ± 0.68a	30.03 ± 4.51a	79.15 ± 7.55a
180	20.93 ± 1.48b	13.38 ± 0.76b	8.35 ± 0.56a	27.66 ± 2.89b	70.32 ± 4.50b

Results are expressed as the mean ± standard deviation ($n = 8$)

$^{a,b}P < 0.05$ demonstrates significant differences between ripening days (two-sample independent *t* test)

[1] Appearance over 30 points

[2] Body and texture over 20 points

[3] Odor over 10 points

[4] Flavor over 40 points

Hydrocarbons

Hydrocarbons have high detection thresholds and therefore their contribution to cheese aroma is much less than other volatile components [54]. Lipid oxidation is seen as a source of hydrocarbons [55]. Styrene, toluene and p-cymene were found to be the most dominant hydrocarbons in Tulum cheeses. Previously, toluene, which is responsible for a nutty flavor note, was reported as predominant hydrocarbon in various cheeses such as Divle Tulum cheese [28], Xinotyri cheese [38], Ibores cheese [27], Torta del Casar cheese [52], and Bouhezza cheese [37]. p-cymene has been identified by some researchers [2, 4, 27, 36]. According to Ozturkoglu-Budak et al. [28], the amounts of hydrocarbons detected in Divle Tulum cheeses were generally low, additionally, there are fluctuations in their amounts during ripening. These authors also reported that the most dominant ones are toluene and styrene in Divle Tulum cheeses. Likewise, Hayaloglu et al. [17] reported that the hydrocarbons found in traditional Gokceada cheese were styrene and toluene, but were detected in low amounts as in our study.

Sensory evaluation

Appearance (e.g., sandy texture, bicolor), body and texture (e.g., distinctive texture, too hard or too soft), odor (e.g., non-defective, moldy, fruity, unpleasant), and taste features (e.g., typical taste, bland taste, sour taste, salty taste) for the sensory characterization of cheeses characteristics were evaluated on the 90th and 180th days of ripening. The scores given by the panelists to other properties except odor were found to be statistically significant during storage and the t-test (two-sample independent t-test) was used to compare this significance. It was determined that the appearance, body, and texture and taste were higher on the 90th day of ripening compared to the 180th day and were statistically significant, while odor was not statistically significantly different from 90 to 180 days. Table 2 shows that higher scores were registered at 90 days of ripening with a total average of 79.15 in the evaluation made over 100 in terms of examining the sensory properties of cheeses. During the ripening period of cheeses, reactions such as lipolysis and proteolysis are occurring [56]. It is known that excessive lipolytic activity is undesirable in cheeses with a long maturation period, and it has also been reported that specific low molecular weight peptide levels that increase as a result of excessive proteolytic activity may cause bitterness [57]. Similarly, Hernández et al. [58] examined the effects of lipolysis and proteolysis on sensory analysis for Idiazabal cheese produced from ewe's raw milk and emphasized that the increasing amount of lipase improved taste and odor intensities, sharp odor, pungent flavor, and bitterness. Based on the lipolysis, the higher occurrence of butanoic acid and octanoic acid responsible for the goatish and waxy aroma notes, and musty aroma note, respectively, could have affected the flavor preference of consumers on day 180. Similarly, the abundance of hexanoic acid, one of the most important components that provide its unique aroma to cheese, on the 90th day compared to 180th may provide the higher total sensory scores on the 90th day. Also, the accumulation of esters and ketones on day 180 may negatively affect the sensory properties.

Conclusion

In this investigation, according to the GC–MS-SPME results, the most dominant groups of volatile compounds have been found to be carboxylic acids (38.38%), esters (27.40%), ketones (20.05%), and alcohols (10.69%). During the whole ripening period, octanoic acid, ethyl acetate, 2-butanone, and isoamyl alcohol were found to be the most abundant acid, ester, ketone, and alcohols, respectively. In the sensory analysis of cheeses, the best results were obtained on the 90th day of ripening. This study provides information about the general characterization of the volatile compound profile of skin bag Tulum cheeses manufactured from Karaman region of Turkey and also valuable knowledge on artisanal Tulum cheese quality regarding appearance, texture, odor, and flavor. The obtained data for the volatile component analysis can allow the production of standardized Tulum cheese from Karaman region and can be considered in quality control of these cheeses. In future studies, peptide profile and lipolytic activity which may be complementary to this study and can be examined during ripening.

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Declarations

Conflict of interest The authors have declared no conflict interest.

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