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## **Research Article**

# Comparison of Some Quality Properties of Erzincan Tulum Cheeses Produced from Raw and Pasteurized Akkaraman Sheep Milk

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# Abstract

In this research, Erzincan Tulum cheese samples were produced in the Dumlu (Erzurum, Turkey) plateaus with the help of the Savak tribe who are the original producers of cheese and were produced in duplicate by using commercial liquid rennet from raw Akkaraman sheep's milk (cheese R) and pasteurized Akkaraman sheep's milk (cheese P). Produced cheeses were filled into the plastic barrels (2 kg), and ripened in a refrigerator at 4±1 °C for 90 days. The differences in the some microbiological (coliforms, Staphylococcus aureus and yeasts & moulds), chemical and sensory properties of the produced cheeses were examined in monthly periods during the ripening period. In this study, the S. aureus counts were < 2 log cfu/g in all Tulum cheese samples during the ripening. In addition, coliforms counts were  $< 1 \log cfu/g$  in samples produced from pasteurized milk (cheese P) during the ripening. During the ripening, the Tulum cheese samples produced from raw milk (cheese R) has higher total coliforms counts according to the Tulum cheese samples produced from pasteurized milk. The R and P cheese samples have statistically similar yeast and mould counts during the ripening. Total coliforms count in R cheese samples decreased continuously during the ripening. The yeast and mould counts in R and P cheese samples decreased also continuously during the ripening. During the ripening, the dry matter, fat, fat in dry matter, protein, ash, salt, salt in dry matter amounts in P cheese samples were determined to be higher according to R cheese samples. However; during the ripening, the pH and % acidity values in the P cheese samples were found to be lower than R cheese samples. In R and P cheese samples; except for pH, examined all chemical properties increased continuously during the ripening. As a result, it was emerged that the cheese variety and the ripening period affected the microbiological and chemical properties. During the ripening; the color and appearance, odor, texture, flavor, saltiness, fatness, bitterness and oxidation, general acceptability scores of R cheese samples were found to be statistically similar with these sensory property scores of P cheese samples. In terms of general acceptability, it can also be emphasized that cheese produced from sheep raw milk is more admired cheese in cheeses ripened for 3 months.

Key words: Erzincan Tulum cheese, pasteurisation, commercial liquid rennet, ripening period, cheese analysis

# Çiğ ve Pastörize Akkaraman Koyun Sütlerinden Üretilen Erzincan Tulum Peynirlerinin Bazı Kalite Özelliklerinin Karşılaştırılması

# Öz

Bu araştırmada, çiğ ve pastörize Akkaraman koyunu sütlerinden ticari sıvı peynir mayası kullanılarak 2 tekerrürlü olarak Erzincan Tulum peynirleri üretilmiştir. Peynirler, peynirin orjinal üreticileri olan Şavaklar yardımıyla Dumlu (Erzurum, Türkiye) yaylalarında üretilmiştir. Üretilen peynirler 2 kg'lık plastik bidonlara basılmış ve buzdolabında (4±1°C) 90 gün olgunlaştırılmıştır. Peynirlerin mikrobiyolojik (koliform bakteri, *Staphylococcus aureus* ile maya ve küf sayıları), kimyasal ve duyusal özelliklerindeki farklılıklar olgunlaşma süresince incelenmiştir. Olgunlaşma süresince peynir örneklerinin tamamında *S. aureus* sayısı (< 2 log kob/g) ile pastörize sütten üretilen Tulum peyniri (P peynir) örneklerinde koliform bakteri bulunmamıştır (< 1 log kob/g). Olgunlaşma süresince, çiğ sütten üretilen Tulum peyniri (R peynir) örneklerinde, P peynirlerden daha fazla koliform bakteri bulunmuştur. R ve P peynir örneklerindeki maya ve küf sayıları olgunlaşma süresince istatistiksel olarak benzer

bulunmuştur. R peynirlerde koliform bakteri sayıları ile R ve P peynir örneklerinde maya ve küf sayıları olgunlaşma süresince azalmıştır. Olgunlaşma süresince, P peynirlerde kurumadde, yağ, kurumaddede yağ, protein, kül, tuz ve kurumaddede tuz miktarları, R peynir örneklerinden yüksek; ancak, pH ve % asitlik değerleri daha düşük bulunmuştur. R ve P peynir örneklerinde, pH hariç, incelenen tüm kimyasal özellikler olgunlaşma süresince artmıştır. Sonuç olarak, ısıl işlem ve olgunlaşma periyodunun peynirlerin mikrobiyolojik ve kimyasal özelliklerini etkilediği ortaya çıkmıştır. R peynir örneklerinin duyusal puanlarının (renk ve görünüş, koku, tekstür, lezzet, tuzluluk, yağlılık, acılık ve oksidasyon ve genel kabul edilebilirlik), olgunlaşma süresince, P peynir örneklerine benzer olduğu görülmüştür (P<0.05). En fazla beğenilen Tulum peynirinin çiğ sütten üretilen ve 3 ay olgunlaştırılan peynir olduğu belirlenmiştir.

Anahtar kelimeler: Erzincan Tulum peyniri, pastörizasyon, ticari sıvı peynir mayası, olgunlaşma periyodu, peynir analizleri

### Introduction

There are more than 150 varieties of cheese in Turkey. The most popular ones of these varieties are the Beyaz, Kaşar and Tulum cheeses (Çakmakçı, 2011). Erzincan Tulum cheese (Şavak cheese) which is the best known among the Tulum cheeses, is the third most widely produced cheese in Turkey (Cakir and Cakmakci, 2018). Erzincan Tulum cheese has a higher economic value compared to these two cheeses (Cakmakci et al., 2008; Cakmakci, 2011). Because; it is also convenient for exportation, as it has a high nutritional value and the fact that it is more expensive than butter increases its economic importance (Cakir and Cakmakci, 2018). Erzincan Tulum cheese is white or cream in colour, has a high-fat content, a buttery flavour, and semi-hard texture and crumbles easily (Çakmakçı, 2011). However, Erzincan Tulum cheese is first cheese of Turkey with a geographical indication status by Patent and Trademark Office in 2000 (TÜRKPATENT, 2000). The manufacturing and ripening processes of Tulum cheese have been extensively reviewed and discussed by Hayaloglu et al. (2007; 2008), Çakmakçı (2011), Cakir et al. (2016) and Cakir and Cakmakci (2018) in detail. Tulum cheese is produced in plateaus around Erzincan, Erzurum, Tunceli, Bingöl and Elazığ (in the East Anatolian regions, Turkey) by the Savak tribe using the milk of Akkaraman sheep between May and September. Tulum cheese is originally ripened in Tulum caves for 3 months or the period could extend up to one year (Cakir et al., 2016). However today, into different sizes plastic hardened barrels of are tightly filled and stored at 2-4 °C.

Studies on Tulum cheese have focused on its chemical and microbiological characteristics and the effects of different packaging materials on its various properties during ripening (Çakmakçı, 2011; Arslaner and Bakırcı, 2016). Sengul and Cakmakci (2003) examined of isolation and identification the lactic acid bacteria of the cheese. Hayaloglu et al. (2007) investigated the microbiology, biochemistry and volatile profiles of Tulum cheese. Cakmakci et al. (2011) studied the proteolysis, sensory properties and gross composition of Tulum cheese ripened in goat's skin or plastic bags. The effects of the addition of black cumin on the detail properties of the Tulum cheese during the ripening period have also been investigated (Cakir et al., 2016; Cakir and Cakmakci, 2018). The fatty acid profile, physicochemical and sensory characteristics were found during the ripening of Tulum cheese produced by using raw and pasteurized goat's milk (Sert et al., 2014).

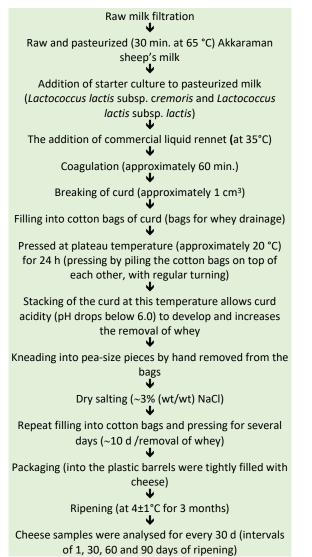
In the original production of cheese, raw sheep milk and home-made calf rennet are used. In recent years, it is known commercial rennet is used in cheese production. In addition, the thoughts of whether cheese can be made from pasteurized milk are also dominant. Therefore, in this research, the effects of the pasteurization and the ripening period on some microbiological, chemical and sensory characteristics of Erzincan Tulum cheeses, which is produced from raw and pasteurized Akkaraman sheep's milk using the commercial liquid rennet and ripened in plastic barrels were investigated.

# Materials and Methods Materials

Raw and pasteurized Akkaraman sheep's milk, salt and commercial liquid rennet used in the production of Erzincan Tulum cheese were provided from Şavak tribe who is producing Tulum cheese and coming to Tortum (Dumlu) plateaus (Erzurum, Turkey) from Elazığ (Turkey). The plastic barrels (2 kg capacity) used in the packaging of cheese were supplied from Çeloğlu Inc. The packaging of the cheeses was carried out in Elazığ (Turkey). The cheeses were stored at 4±1°C were tested during the ripening period.

#### **Cheese-making**

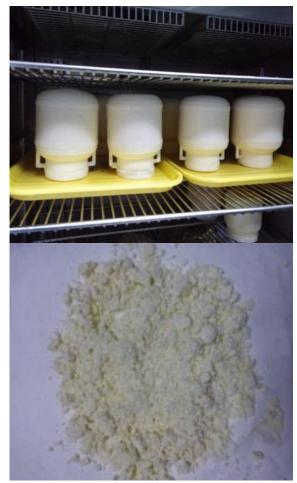
Erzincan Tulum cheese production is shown in Fig. 1. The cheese making was made in duplicate. An aspect from Erzurum (Dumlu, Tortum) Plateaus is shown in Fig. 2. The cheese samples in plastic barrel is shown in Fig. 3.



**Figure 1.** A flow sheet for the production of Erzincan Tulum cheese



**Figure 2.** A photograph from Erzurum, Turkey (Dumlu/Tortum) Plateaus



**Figure 3.** Erzincan Tulum cheese samples in plastic barrel and unpacked cheese

#### Sampling

The cheeses were sampled from the separately plastic barrels (2 kg capacity) at each sampling time (1, 30, 60, 90 d), but before taking samples the surface section of the cheese samples (approximately 0.5 to 1.0 cm) was discarded. For microbiological analysis, 100 g samples were taken in sterile glass jars and 200 g for chemical analysis. The cheese remaining in the plastic barrels were used for sensory analysis. For microbiological analysis, into 2 sterile glass jars (1 of which were stored at -20 °C as a backup sample) were collected approximately 100 g separately, and about 200 g samples for chemical analysis. The cheese remaining in the plastic barrels was used for sensory analysis.

#### **Microbiological analysis**

For microbiological analysis, 10 g of Tulum cheese was weighed and dispersed aseptically in 90 mL of saline peptone water that included 0.85% NaCl with 0.1% peptone. The mixture was homogenized (8 strokes per second) in a sterile polyethylene bag using a Stomacher (Mayo HG400 Stomacher, Italy) for 2 min. Serial dilutions were made in saline peptone water (ISO 6887) (Harrigan, 1998), and all tests were performed in duplicate. Total coliforms were enumerated on Violet Red Bile Agar (VRBA, Merck 1.01406, Germany) at 35-37 °C for 48 h (Harrigan, 1998), yeasts and moulds on Potato Dextrose Agar (PDA, Merck 1.10130, Darmstadt, Germany) at 25 °C for 5-7 days (Harrigan, 1998), and *Staphylococcus aureus* on Baird-Parker Agar (BPA, Merck 1.05406, Germany) at 37 °C for 24 h (Harrigan, 1998).

#### **Chemical analysis**

Tulum cheese samples were tested during the ripening period at the  $1^{st}$ ,  $30^{th}$ ,  $60^{th}$ , and  $90^{th}$ days. Dry matter amount was examined by the oven-drying method at 105 °C (IDF, 1982), fat amount by the method of Gerber, ash and salt amounts according to the methods defined by Kurt et al. (2007). Titratable acidity (lactic acid, %) was measured as described by AOAC (1995). For pH measurement, grated cheese sample (10 g) was mixed uniformly with distilled water (15 mL), and the pH of the resultant slurry was measured using composite electrode, a digital pH meter (pH 211, Microprocessor pH Meter, Hanna Inst., Italy) (Savello et al., 1989). Tulum cheese samples were examined in duplicate for determination of total nitrogen (TN) amount by the micro-Kjeldahl method (IDF, 1993), and the protein amount was calculated (TN % × 6.38).

### Sensory analysis

Erzincan Tulum cheese samples were evaluated using a sensory scale according to the methods of Bodyfelt et al. (1988) and Altug (1993) during the ripening period. Sensory criteria were developed taking the properties of the Tulum cheese into consideration. Thirty panelists who were familiar with Erzincan Tulum cheese and were academic staff (ages were between 20 and 60) at the Department of Food Engineering (Atatürk University, Erzurum, Turkey and other consumers) staffed in the evaluation of cheeses. Each panelist evaluated the cheeses for the eight sensory properties including color or appearance, odor, texture, flavor, saltiness, fatness, bitterness and oxidation and general acceptability. All sensory properties were graded from 1-9 (1: poor, 9: excellent) on score scales. Panelists were also instructed to cleanse their palates between samples by using water and bread.

### **Statistical analysis**

The experiments were conducted in a completely randomised design in a factorial arrangement: two treatments of cheese (R and P),

four ripening periods (1<sup>st</sup>, 30<sup>th</sup>, 60<sup>th</sup> and 90<sup>th</sup> days) and two replicates. All data were analyzed statistically using SPSS 17.0 programme (SPSS Inc., Chicago, IL, USA). Analysis of variance (ANOVA) and Duncan's Multiple Range Test were used to determine significant differences among results.

# **Results** and **Discussion** Microbiological characteristics

The evolution of the different microorganisms throughout the cheese samples ripening period (0-90 days) is presented in Table 1. The cheese variety and the ripening period has statistically a significant effect on the total coliforms counts (P < 0.05) (Table 1).

In the Tulum cheese sample produced from raw milk (R), the maximum and minimum levels of total coliforms count ranged from 3.10 log cfu/g (the initial value of the ripening period, fresh cheese) to 1.67 log cfu/g (the final value of the ripening period), respectively (Table 1). The total coliforms count reported in the R sample were lower than the values determined by Hayaloglu et al. (2007) for Tulum cheese, were higher than the values determined by Ceylan et al. (2007) and for Tulum cheeses and Cakmakci et al. (2012) for mouldy Civil cheese, were in accordance with the values determined by Cakir and Cakmakci (2018) for Tulum cheese and Hayaloglu and Kirbag (2007) for mould-ripened Kuflu cheese. Additionally, in the Tulum cheese sample produced from raw milk (R), total coliforms count decreased continuously during the ripening period (Table 1). Similar results were also found by Ceylan et al. (2007), Hayaloglu et al. (2007), Cakir and Cakmakci (2018) and Tomar et al. (2018) for Tulum cheeses. Lactic acid bacteria and moulds have an inhibitory effect on coliform group bacterial growth and, it is also thought that the coliform group bacteria count decreased with the decrease in water activity due to moisture loss during ripening (Martin et al., 2016).

In the cheese sample produced from pasteurized milk (P), total coliforms counts were < 1 log cfu/g during the ripening period (Table 1). This result may have resulted from the diligence shown for pasteurization of milk and subsequently to prevent contamination during the cheese production stages. Same results were also found by Ceylan et al. (2007) for Tulum cheese. The total coliforms count reported in the P sample were lower than the results determined by Cakir and Cakmakci (2018) and Hayaloglu et al. (2007) for Tulum cheeses, Cakmakci et al. (2012) for mouldy Civil cheese, Hayaloglu and Kirbag (2007) for mouldripened Kuflu cheese. These results may be caused by the original production of the cheese from raw sheep's milk.

Pasteurisation has not a statistically significant effect on the yeast and mould counts (P > 0.05) (Tables 1). The R and P cheese samples have statistically similar yeast and mould counts during the ripening period. In this research, the highest and lowest yeast and mould counts were recorded in the R (6.65 log cfu/g, the initial value) and P (5.91 log cfu/g, the final value) the cheeses, respectively (Table 1). The yeast and mould counts reported in this research were higher than the values determined by Cakir and Cakmakci (2018), Hayaloglu et al. (2007), Ceylan et al. (2007), Tarakci et al. (2005) for Tulum cheeses; however, were lower than the results determined by Cakmakci et al. (2012) for mouldy Civil cheese, Cakmakci et al. (2014) for mould-ripened Civil cheese, Hayaloglu and Kirbag (2007) for mould-ripened Kuflu cheese, Gobbetti et al. (1997) for Gorgonzola cheese.

The ripening period has statistically a significant effect on the yeast and mould counts (P < 0.05) (Table 1). The yeast and mould counts in all cheese samples decreased continuously during the ripening period (Table 1). Similar result was also found by Cakir and Cakmakci (2018) for Tulum cheeses.

There is no limitation on the count of coliform group bacteria, yeast and moulds in Tulum Cheese Standard (TS 3001) (TS-3001, 2016) and in Turkish Food Codex Regulation-Microbiological Criteria Communique' (2009/68) about Tulum cheese. However, it has been reported that the count of Enterobacteriaceae family can have a highest of 10<sup>3</sup> cfu/g. According to the Food Safety Centre (2014), the count of entrances of ripened cheeses can be accepted in the range of  $10^2 - 10^4$ cfu/g. The count of *Escherichia coli* can be up to  $10^2$ cfu/g, but the decision is not valid to cheeses made from raw milk. According to Scientific Criteria to Ensure Safe Food (2003), cheeses made from raw milk and from thermified milk (Directive 92/46/EEC) show that up to  $10^4$  cfu/g count *E. coli* can be found.

S. aureus is a microorganism showing high sensitivity to all applications for inhibition of microorganisms including especially heat treatment. Non-heat treated foods, mastitis milks (Charlier et al., 2009), dirty hands, insufficient sanitized instrument equipment and sewerage are contamination sources of S. aureus. In this research, the S. aureus counts were < 2 log cfu/g in all Tulum cheese samples during the ripening period; namely, S. aureus was not detected in any samples during the ripening period (Table 1). Same results were also found by Tarakci et al. (2005), Cakir and Cakmakci (2018) for Tulum cheeses and Cakmakci et al. (2012) for mouldy Civil cheese. In this research,

the *S. aureus* counts reported in the cheese sample were lower than the results determined by Hayaloglu and Kirbag (2007) for mould-ripened Kuflu cheese. According to the TS 3001 (2016) and the Turkish Food Codex Regulation-Communique' on Microbiological Criteria (2009/68), the count of *S. aureus* of all cheeses excluding melted cheese can be accepted as  $10^2$  cfu/g. The storage temperature (2-4 °C) and salt concentration (2.5-3%) may be limiting factors for the growth of these microorganisms (Cakir and Cakmakci, 2018)

## **Chemical characteristics**

The mean and standard deviation for the dry matter, fat, fat-in-dry matter (FDM), protein, ash, salt, salt-in-dry matter (SDM), pH and titratable acidity of Tulum cheese samples during ripening (1 and 90 days) are summarized in Table 2.

In this study, pasteurisation process determined to have statistically a significant effect on some components of the cheese (P<0.05) (Table 2). According to this, the P sample produced from pasteurized milk has higher dry matter, fat, fat in dry matter, protein, ash, salt, salt in dry matter values compared to the R sample produced from raw milk. However, P sample has lower pH and % acidity values compared to the R cheese (Table 2). In the study made by Sert et al. (2014), similarly, the Tulum cheese produced from pasteurized milk has higher dry matter, fat, fat in dry matter, ash, salt, salt in dry matter values compared to the Tulum cheese from raw milk. In the same research, Tulum cheese produced from raw milk has higher % acidity values, and lower pH values, compared to the Tulum cheese produced from pasteurized milk.

In this study, the highest and lowest values of the dry matter, fat, fat-in-dry matter (FDM), protein, ash, salt, salt-in-dry matter (SDM) contents were recorded in the P cheese at 90<sup>th</sup> days of (57.66-31.63-54.85-3.186-20.33-4.76ripening 3.22-5.59%, the final value) and R cheese at 1<sup>st</sup> day of ripening (55.51-30.25-54.50-3.025-19.30-4.43-3.04-5.48%, the initial value), respectively (Table 2). In this research; the dry matter contents of samples were higher than the results determined by Sert et al. (2014), Cakmakci et al. (2011), Hayaloglu et al. (2007) for Tulum cheeses, Cakmakci et al. (2012) for mouldy Civil cheese, Cakmakci et al. (2014) for mould-ripened Civil cheese, were lower than the results reported by Cakir and Cakmakci (2018), Cakir et al. (2016) for Tulum cheeses. The fat contents of the cheese samples were higher than the results determined by Sert et al. (2014), Cakmakci et al. (2011), Ceylan et al. (2007) for Tulum cheeses. The FDM contents of the cheese samples were higher than the results determined by Sert et al. (2014) for Tulum cheeses, were lower than the results

determined by Hayaloglu et al. (2007), Cakir and Cakmakci (2018), Cakir et al. (2016) for Tulum cheeses. Protein contents of samples were lower than the results determined by Ceylan et al. (2007), Cakir and Cakmakci (2018), Cakir et al. (2016) for Tulum cheeses, were higher than the results determined by Hayaloglu et al. (2007) for Tulum cheese. The ash contents of Tulum cheese samples were higher than the results determined by Cakir and Cakmakci (2018), Cakir et al. (2016) for Tulum cheeses, were lower than the results determined by Ceylan et al. (2007) and Sert et al. (2014) for Tulum cheeses. The salt contents of Tulum cheese samples were lower than the results determined by Ceylan et al. (2007), Sert et al. (2014) for Tulum cheeses, were higher than the values determined by Cakir and Cakmakci (2018) for Tulum cheese, were in agreement with the results determined by Cakir et al. (2016) for Tulum cheese. The salt-in-dry matter (SDM) contents of the cheeses were higher than the results determined by Cakir and Cakmakci (2018), Cakir et al. (2016), Cakmakci et al. (2011) for Tulum cheeses, were lower than the results determined by Sert et al. (2014) for Tulum cheese.

According to the TS-3001 (2016), the moisture amount of Erzincan Tulum cheese is restricted to at highest 45%. The moisture amount was under 45%, as defined in the regulations for Erzincan Tulum cheese according to the Turkish Food Codex (Anonymous, 2015). The dry matter amount was not under 60%, as is reported in the regulations for Erzincan Tulum cheese in the Turkish Food Codex (Anonymous, 2015). In addition, the FDM contents of Tulum cheese samples in this study were  $\geq$  45% (v/w); therefore, these Tulum cheeses could be classified as full-fat Tulum cheeses according to the regulations for Erzincan Tulum cheeses according to the regulations for Erzincan Tulum cheeses (Anonymous, 2015).

The maximum and minimum pH values were found in the R (5.02, at 1<sup>st</sup> and 90<sup>th</sup> days of ripening) and P (4.63, at 90<sup>th</sup> days of ripening) cheese samples, respectively (Table 2). In this study; the pH values of the samples were in accordance with the values determined by Cakir and Cakmakci (2018), Cakir et al. (2016), Cakmakci et al. (2011) and Tomar et al. (2018) for Tulum cheeses..

Type of cheese (Treatment)	Ripening time (Days)	Total coliforms	S. aureus	Yeasts & moulds
	1	3.10±0.14	<2	6.65±0.14
	30	2.93±0.04	<2	6.28±0.12
R	60	2.37±0.23	<2	6.01±0.23
	90	1.67±0.25	<2	5.38±0.20
	Average	2.52±0.61 <b>a</b>	<2	6.08±0.51 <b>a</b>
	1	<1	<2	6.55±0.21
(Treatment) R P	30	<1	<2	6.37±0.11
Р	60	$3.10\pm0.14$ <2 $6.65\pm0.2$ $2.93\pm0.04$ <2	5.51±0.35	
	90		5.21±0.28	
	Average	<1 <b>b</b>	<2 <2 <2 <2 <2 <2 <2 <2 <2 <2 <2 <2 <2 <	5.91±0.63 <b>a</b>
	1	1.55±1.79 <b>a</b>	<2	6.60±0.16 <b>a</b>
(Treatment) R P	30	1.46±1.69 <b>a</b>	<2	6.32±0.11 <b>a</b>
		<2	5.76±0.38 <b>b</b>	
	90	0.84±0.98 <b>c</b>	<2	5.30±0.22 <b>c</b>
	Average	1.26±1.37	<2	5.99±0.56

 Table 1. Microbiological properties of Erzincan Tulum cheese samples during the ripening period (log cfu/g)

R: Tulum cheese produced from raw Akkaraman sheep's milk; P: Tulum cheese produced from pasteurized Akkaraman sheep's milk. \*Averages of the same column values (each section separately) by the same letter did not differ significantly from Duncan's multiple range tests at 5% significance. (a-c) Mean ± SD, values followed by the same letters within a column are significantly different at P<0.05.

In general, a decrease in pH is waited to consist at the first stages of ripening because of the metabolism of remaining lactose to lactic acid, followed by an increase in pH depending on the variety of cheese (Fox et al., 1999; Sert et al., 2014). The pH of mould-ripened cheeses increases during the ripening because of the catabolism of lactic acid and deamination of amino acids by moulds (Hayaloglu et al., 2008). The pH values of the samples in this research were lower than the pH values for blue-type cheeses, including Cakmakci et al. (2012, 2014) for mouldy Civil cheeses, Hayaloglu et al. (2008) and Hayaloglu and Kirbag (2007) for mould-ripened Kuflu cheeses, Gobbetti et al. (1997) for Gorgonzola cheese, Seratlic et al. (2011) for Gorgonzola type cheese, Madkor et al. (1987) for commercial Stilton cheese, Galli et al. (2016) for Camembert-type cheese. The high pH in blue-type

cheeses has been connected with the catabolism of lactic acid in cheese by mycoflora (Cakmakci et al., 2012; Hayaloglu et al., 2008).

The maximum and minimum values of acidity were found in R (1.395%, the final value at 90<sup>th</sup> days of ripening) and P (0.963%, the initial value at 1<sup>st</sup> day of ripening), respectively (Table 2). The acidity values of Tulum cheese samples were in accordance with the values determined by Hayaloglu et al. (2007), Cakmakci et al. (2011) Sert et al. (2014) for Tulum cheeses, were higher than the results reported by Cakir et al. (2016), Cakir and Cakmakci (2018), Tomar et al. (2018) for Tulum cheeses, Cakmakci et al. (2012, 2014) for mouldy Civil cheese, Hayaloglu et al. (2008) for mould-ripened Kuflu cheeses, and were lower than the results determined by Ceylan et al. (2007) for Tulum cheese.

The ripening period has statistically a significant effect on the other all components except for the fat in dry matter of the cheese samples (P<0.05) (Table 2). The dry matter, fat, fat in dry matter, protein, ash, salt, salt in dry matter, % acidity values of the samples showed continuous an increase during the ripening period (until the end of ripening) (Table 2). Thus, ripening time was decisive for microbiological and physicochemical properties of sheep milk cheese characteristics. Similar increases in the studies made by Ceylan et al. (2007), Havaloglu et al. (2007), Sert et al. (2014), Cakir and Cakmakci (2018) for Tulum cheeses were also determined during the ripening period. These increases in the chemical parameters were due to the decrease in moisture during the ripening. The decrease in moisture is caused by microbial multiplication and the acid development that occurs during the ripening, as well as by syneresis of the cheeses (Cakir and Cakmakci 2018; Moreira et al., 2020). Other factors causing the decrease in moisture may be the reduced hydration of casein as the pH reaches the isoelectric point (Moreira et al., 2020), and also the evaporation of moisture from cheese through the pores of the ripening material (Fox et al. 1999; Ceylan et al. 2007).

# Sensory characteristics

The sensory properties of the cheese samples during the ripening period are presented in Table 3. The cheese variety and in general the ripening period had not statistically a significant effect on the sensory properties of cheese samples (P>0.05) (Table 3).

During ripening, the sensory properties scores (color and appearance, odor, texture, flavor, saltiness, fatness, bitterness and oxidation, general acceptability) of the cheese samples produced from raw milk were statistically similar to the sensory properties scores of the cheese samples produced from pasteurized milk (P> 0.05) (Table 3).

According to the results of the sensory analysis, during the ripening period, the sensory characteristics of samples (the general acceptability of samples) were not found statistically different and the cheese samples were scored with good marks (high points) (7-8 out of 9) (Table 3). Additionally, the highest and lowest values of the color and appearance, odor, texture, flavor, saltiness, fatness, bitterness and oxidation, general acceptability scores of the Tulum cheese samples ranged from 8.58 to 7.82, from 8.04 to 7.25, from 8.25 to 7.57, from 8.26 to 7.84, from 8.65 to 7.95, from 8.20 to 7.75, from 8.40 to 7.46 and from 8.40 to 7.85, respectively (Table 3). In general, all sensory properties scores of the Tulum cheese samples were higher than the sensory properties scores determined by Ceylan et al. (2007), Sert et al. (2014), Cakir et al. (2016) for Tulum cheeses, Cakmakci et al. (2013) for mould-ripened Civil cheese, and were in accordance with the sensory properties scores determined by Cakmakci et al. (2011) for Tulum cheese.

# Conclusions

In this study; pasteurization of milk, the Tulum cheese sample produced from pasteurized milk have higher dry matter, fat, fat in dry matter, protein, ash, salt and salt in dry matter values compared to the Tulum cheese sample produced from raw milk. Also, it can be said that the dry matter amounts increased in Tulum cheese samples due to moisture loss during the ripening period. Depending on increase in the dry matter amounts of Tulum cheese samples; fat, fat in dry matter, protein, ash, salt and salt in dry matter values increased during the ripening period.

Erzincan Tulum cheese, which is considered as our most important traditional cheese. In this study, it was determined that in the production of Tulum cheese, if starter culture is used after pasteurization of milk, it does not make a significant difference from the cheese produced from raw milk in terms of sensory properties examined. However, aroma-active volatile compounds effective in cheese flavor should also be determined. It can be stated that Erzincan Tulum cheese can be consumed safely and with pleasure after 3 months, even if it is traditionally produced (from raw sheep milk) in plateaus and under controlled conditions.

Type of cheese (Treatment)	Ripening time (Days)	Dry matter (%)	Fat (%)	Fat in dry matter (%)	Protein (%)	Ash (%)	Salt (%)	Salt in dry matter (%)	рН	Acidity (%)
R	1	55.51±0.06	30.25±0.00	54.50±0.06	19.30±0.10	4.43±0.04	3.04±0.00	5.48±0.01	5.02±0.01	0.981±0.013
	30	55.71±0.19	30.38±0.18	54.53±0.13	19.46±0.08	4.48±0.04	3.07±0.04	5.51±0.06	4.89±0.01	1.161±0.013
	60	55.96±0.09	30.63±0.18	54.73±0.23	19.56±0.02	4.52±0.02	3.10±0.00	5.54±0.01	4.85±0.01	1.278±0.025
	90	56.18±0.03	30.75±0.00	54.74±0.02	19.73±0.04	4.56±0.04	3.13±0.04	5.57±0.07	5.02±0.04	1.395±0.013
	Average	55.84±0.28 <b>a</b>	30.50±0.23 <b>a</b>	54.62±0.15 <b>a</b>	19.51±0.17 <b>a</b>	4.49±0.06 <b>a</b>	3.09±0.04 <b>a</b>	5.52±0.05 <b>a</b>	4.94±0.09 <b>a</b>	1.204±0.164 <b>a</b>
	1	56.96±0.05	31.13±0.18	54.65±0.26	20.02±0.05	4.63±0.04	3.13±0.04	5.50±0.06	4.66±0.04	0.963±0.013
	30	57.15±0.02	31.25±0.00	54.69±0.02	20.11±0.04	4.67±0.03	3.16±0.00	5.53±0.00	4.79±0.00	1.062±0.025
Р	60	57.31±0.06	31.38±0.18	54.76±0.25	20.22±0.08	4.71±0.04	3.19±0.04	5.57±0.06	4.69±0.01	1.161±0.013
	90	57.66±0.25	31.63±0.18	54.85±0.07	20.33±0.06	4.76±0.05	3.22±0.00	5.59±0.02	4.63±0.01	1.278±0.025
	Average	57.27±0.29 <b>b</b>	31.34±0.23 <b>b</b>	54.73±0.16 <b>a</b>	20.17±0.13 <b>b</b>	4.69±0.06 <b>b</b>	3.18±0.04 <b>b</b>	5.54±0.05 <b>a</b>	4.69±0.07 <b>b</b>	1.116±0.126 <b>b</b>
Treatment average	1	56.23±0.84 <b>a</b>	30.69±0.52 <b>a</b>	54.57±0.18 <b>a</b>	19.66±0.42 <b>a</b>	4.53±0.12 <b>a</b>	3.09±0.06 <b>a</b>	5.49±0.04 <b>a</b>	4.84±0.21 <b>a</b>	0.972±0.015 <b>a</b>
	30	56.43±0.84 <b>a</b>	30.81±0.52 <b>ab</b>	54.61±0.12 <b>a</b>	19.78±0.38 <b>b</b>	4.57±0.12 <b>ab</b>	3.12±0.06 <b>ab</b>	5.52±0.03 <b>ab</b>	4.84±0.06 <b>a</b>	1.112±0.059 <b>b</b>
	60	56.63±0.78 <b>b</b>	31.00±0.46 <b>b</b>	54.74±0.19 <b>a</b>	19.89±0.38 <b>c</b>	4.61±0.11 <b>b</b>	3.15±0.06 <b>b</b>	5.55±0.04 <b>ab</b>	4.77±0.09 <b>b</b>	1.220±0.070 <b>c</b>
	90	56.92±0.86 <b>c</b>	31.19±0.52 <b>c</b>	54.79±0.08 <b>a</b>	20.03±0.35 <b>d</b>	4.66±0.12 <b>c</b>	3.18±0.06 <b>c</b>	5.58±0.04 <b>b</b>	4.82±0.23 <b>a</b>	1.337±0.070 <b>d</b>
	Average	56.55±0.79	30.92±0.49	54.68±0.16	19.84±0.37	4.59±0.12	3.13±0.06	5.53±0.05	4.82±0.15	1.160±0.148

Table 2. The changes in some components of Erzincan Tulum cheese samples during the ripening period

R: Tulum cheese sample produced from raw Akkaraman sheep's milk; P: Tulum cheese sample produced from pasteurized Akkaraman sheep's milk. \*Averages of the same column values (each section separately) by the same letter did not differ significantly from Duncan's multible range tests at 5% significance. (a-d) Mean ± SD, values followed by the same letters within a column are significantly different at P<0.05.

**Table 3.** The changes in sensory characteristics of Erzincan Tulum cheese samples during the ripening period(1: poor, 9: like)

Type of cheese (Treatment)	Ripening time (Days)	Color and appearance	Odor	Texture	Flavor	Saltiness	Fatness	Bitterness and oxidation	General acceptability
	30	8.58±0.60	7.63±0.18	8.25±0.35	8.00±0.00	8.38±0.18	7.88±0.18	8.40±0.14	8.23±0.04
	60	8.22±0.40	8.04±0.66	7.72±0.40	8.05±0.07	8.07±0.10	8.00±0.35	7.97±0.05	8.04±0.05
R	90	8.04±0.06	7.74±0.37	7.84±0.23	8.06±0.08	8.06±0.08	8.15±0.21	7.46±0.64	7.97±0.04
	Average	8.28±0.41 <b>a</b>	7.80±0.39 <b>a</b>	7.93±0.36 <b>a</b>	8.04±0.05 <b>a</b>	8.17±0.19 <b>a</b>	8.01±0.24 <b>a</b>	7.94±0.52 <b>a</b>	8.08±0.12 <b>a</b>
	30	8.50±0.71	7.25±0.35	8.00±0.71	8.05±0.07	8.33±0.25	7.75±0.35	8.38±0.18	8.40±0.14
_	60	8.32±0.25	7.79±0.30	7.98±0.74	8.26±0.35	8.65±0.49	8.05±0.21	7.93±0.10	8.11±0.13
Р	90	7.82±0.17	7.42±0.11	7.57±0.62	7.84±0.20	7.95±0.08	8.20±0.28	7.69±0.26	7.85±0.21
	Average	8.21±0.47 <b>a</b>	7.49±0.33 <b>a</b>	7.85±0.58 <b>a</b>	8.05±0.26 <b>a</b>	8.31±0.40 <b>a</b>	8.00±0.30 <b>a</b>	8.00±0.35 <b>a</b>	8.12±0.28 <b>a</b>
	30	8.54±0.54 <b>a</b>	7.44±0.31 <b>a</b>	8.13±0.48 <b>a</b>	8.03±0.05 <b>a</b>	8.35±0.18 <b>a</b>	7.81±0.24 <b>a</b>	8.39±0.13 <b>a</b>	8.31±0.13 <b>a</b>
Treatment average	60	8.27±0.28 <b>a</b>	7.91±0.44 <b>a</b>	7.85±0.51 <b>a</b>	8.15±0.24 <b>a</b>	8.36±0.44 <b>a</b>	8.03±0.24 <b>a</b>	7.95±0.07 <b>ab</b>	8.07±0.09 <b>b</b>
	90	7.93±0.16 <b>a</b>	7.58±0.29 <b>a</b>	7.70±0.41 <b>a</b>	7.95±0.17 <b>a</b>	8.00±0.09 <b>a</b>	8.18±0.21 <b>a</b>	7.57±0.42 <b>b</b>	7.91±0.14 <b>b</b>
	Average	8.25±0.42	7.64±0.38	7.89±0.46	8.04±0.18	8.24±0.31	8.00±0.26	7.97±0.42	8.10±0.21

R: Tulum cheese sample produced from raw Akkaraman sheep's milk; P: Tulum cheese sample produced from pasteurized Akkaraman sheep's milk. \*Averages of the same column values (each section separately) by the same letter did not differ significantly from Duncan's multible range tests at 5% significance. (a-b) Mean ± SD, values followed by the same letters within a column are significantly different at P<0.05.

Although Erzincan Tulum cheese is a type of cheese ripening for 3 months or more, it can also be said that if it is produced from pasteurized milk with the addition of starter culture, it can be safely consumed at every stage from production. In terms of general acceptability, it can also be emphasized that cheese produced from raw milk is more admired cheese in cheeses matured for 3 months.

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**Conflict of Interest Statement:** The manuscript's authors declare that, they do not have any conflict of interest.

# **Researchers' Contribution Rate Statement**

**Summary:** The authors declare that, they have contributed equally to the manuscript.

## References

- Altuğ, T., 1993. Duyusal Test Teknikleri. Ege Üniversitesi Mühendislik Fakültesi Ders Kitapları Yayınları No: 28, 56 s, İzmir.
- Anonymous, 2015. Turkish food codex, for cheese. Number 2015/6. Official Journal, 29261, Ankara, Turkey.
- AOAC, 1995. Official Methods of Analysis, Vol. 2., 16th ed. AOAC International, Arlington, VA, pp. 503-515.
- Arslaner, A., Bakırcı, İ., 2016. Effect of milk type, pasteurization and packaging materials on some physicochemical properties and free fatty acid profiles of Tulum cheese. Akademik Gıda, 14(2), 98-104.
- Bodyfelt, F.W., Tobias, J. and Trout, G.M., 1988. The Sensory evoluation of dairy products. Published by Van Nastrand Reinhold, p. 598, New York, USA.
- Cakir, Y. and Cakmakci, S., 2018. Some microbiological, physicochemical and ripening properties of Erzincan Tulum cheese produced with added black cumin (*Nigella sativa* L.). Journal of Food Science and Technology, 55(4), 1435-1443.
- Cakir, Y., Cakmakci, S. and Hayaloglu, A. A., 2016. The effect of addition of black cumin (*Nigella sativa* L.) and ripening period on proteolysis, sensory properties and volatile profiles of Erzincan Tulum (Şavak) cheese made from

raw Akkaraman sheep's milk. Small Ruminant Research, 134, 65-73.

- Cakmakci, S., Dağdemir, E., Hayaloğlu, A.A., Gürses, M., Gündoğdu, E., 2008. Influence of ripening container on the lactic acid bacteria population in Tulum cheese. World Journal of Microbiology and Biotechnology, 24, 293-299.
- Cakmakci, S., Gürses, M., Gündoğdu E., 2011. The effect of different packaging materials on proteolysis, sensory scores and gross composition of Tulum cheese. African Journal of Biotechnology, 10(21), 4444-4449.
- Cakmakci, S., Gündoğdu, E., Hayaloğlu, A.A., Dağdemir, E., Gürses, M., Çetin, B., Tahmas-Kahyaoğlu, D., 2012. Chemical and microbiological status and volatile profiles of mouldy Civil cheese, a Turkish mouldripened variety. International Journal of Food Science and Technology, 47(11), 2405-2412.
- Cakmakci, S., Dağdemir, E., Hayaloğlu, A.A., Gürses, M., Çetin, B., Tahmas-Kahyaoğlu, D., 2013. Effect of *Penicillium roqueforti* and incorporation of whey cheese on volatile profiles and sensory characteristics of mould-ripened Civil cheese. International Journal of Dairy Technology, 66 (4), 512-526.
- Cakmakci, S., Hayaloğlu, A.A., Dağdemir, E., Çetin, B., Gürses, M., Tahmas-Kahyaoğlu, D., 2014. Effects of *Penicillium roqueforti* and whey cheese on gross composition, microbiology and proteolysis of mould-ripened Civil cheese during ripening. International Journal of Dairy Technology, 67 (4), 594-603.
- Centre for Food Safety, 2014. Microbiological guidelines for food (for ready-to-eat food in general and specific food items) August 2014 (revised). Risk Assessment Section Centre for Food Safety Food and Environmental Hygiene Department 43/F, Queensway Government Offices, 66 Queensway, Hong Kong.
- Ceylan, Z. G., Çağlar, A., Çakmakçı, S., 2007. Some physicochemical, microbiological and sensory properties of Tulum cheese produced from ewe's milk via a modified method. International Journal of Dairy Technology, 60 (3), 191-197.
- Charlier, C., Cretenet, M., Even, S. and Le Loir, Y., 2009. Interactions between *Staphylococcus aureus* and lactic acid bacteria: An old story with new perspectives. International Journal of Food Microbiology, 131, 30-39.
- Çakmakçı, S., 2011. Türkiye Peynirleri (19. Bölüm: 585-614). In: Peynir Biliminin Temelleri. (Editörler: A.A. Hayaloğlu ve B. Özer), ISBN:

978-605-87976-1-1, SİDAS Medya Ltd. Şti., 643 s, İzmir.

- Fox, P.F., Law, J., McSweeney, P.L.M., Wallace, J., 1999. Biochemistry of cheese ripening. In: Fox PF (ed) Cheese: chemistry, physics and microbiology, vol 1. Aspen Publishers, Gaithersburg, pp 389-438.
- Galli, B.D., Martin, J.G.P., Silva, P.P.M., Porto, E., Spoto, M.H.F., 2016. Sensory quality of Camembert-type cheese: Relationship between starter cultures and ripening molds. International Journal of Food Microbiology, 234, 71-75.
- Gobbetti, M., Burzigotti, R., Smacchi, E., Corsetti, A., De Angelis, M., 1997. Microbiology and biochemistry of Gorgonzola cheese during ripening. International Dairy Journal, 7, 519-529.
- Harrigan, W.F., 1998. Laboratory Methods in Food Microbiology. Academic Press, San Diego, USA, p. 532.
- Hayaloglu, A.A. and Kirbag, S., 2007. Microbial quality and presence of moulds in Kuflu cheese. International Journal of Food Microbiology, 115, 376-380.
- Hayaloglu, A.A., Çakmakçı S., Brenchany K.C., Deegan K.C. and McSweeney P.L.H., 2007. Microbiology, biochemistry and volatile composition of Tulum cheese ripened in goat's skin or plastic bags. Journal of Dairy Science, 90 (3), 1102-1121.
- Hayaloglu, A.A., Brenchany, K.C., Deegan, K.C. and McSweeney, P.L.H., 2008. Characterization of the chemistry, biochemistry and volatile profile of Kuflu cheese, a mould-ripened variety. LWT - Food Science and Technology, 41, 1323-1334.
- IDF (International Dairy Federation), 1982. Determination of the total solid content (cheese and processed cheese). In: IDF Standard 4A. IDF, Brussels, Belgium.
- IDF (International Dairy Federation), 1993. Determination of the nitrogen content (Kjeldahl method) and calculation of the crude protein content. In: Standard Method 20B: Milk, IDF, Brussels, Belgium.
- Kurt, A., Cakmakci, S., and Caglar, A., 2007. Süt ve Mamülleri Muayene Analiz Metotları Rehberi. Atatürk Universitesi Ziraat Fakültesi Yayınları No: 257, 398 p, Erzurum, Turkey (In Turkish).
- Madkor, S., Fox, P.F., Shalabi, S.I., Metwalli, N.H., 1987. Studies on the ripening of Stilton cheese: proteolysis. Food Chemistry, 25, 13-29.
- Martin, N.H., Trmcic, A., Hsieh, T., Boor, K.J., Wiedmann, M., 2016. The evolving role of

coliforms as indicators of unhygienic processing conditions in dairy foods. Frontiers in Microbiology, 7, 1-8. Article: 1549 doi: 10.3389/fmicb.2016.01549

- Moreira, R.V., Costa, M.P., Frasao, B.S., Sobral, V.S., Cabral, C.C., Rodrigues, B.L., Mano, S.B., Conte-Junior, C.A., 2020. Effect of ripening time on bacteriological and physicochemical goat milk cheese characteristics. Food Science and Biotechnology, 29, 459–467. https://doi.org/10.1007/s10068-019-00682w
- Savello, P.A., Ernstrom, C.A., Kalab, M., 1989. Microstructure and meltability of model process cheese made with rennet and acid casein. Journal of Dairy Science, 72, 1-11.
- Scientific Criteria to Ensure Safe Food, 2003. The National Academies Press. https://doi.org/10.17226/10690.
- Sengul, M., Cakmakci, S., 2003. Characterization of natural isolates of lactic acid bacteria from Erzincan (Savak) Tulum cheese. Milchwissenschaft-Milk Science International, 58(9-10), 510-513.
- Seratlic, S., Miloradovic, Z.N., Radulovic, Z.T., Macej, O.G., 2011. The effect of two types of mould inoculants on the microbiological composition, physicochemical properties and protein hydrolysis in two Gorgonzolatype cheese varieties during ripening. International Journal of Dairy Technology, 64, 408-416.
- Sert, D., Akın, N., Aktümsek, A., 2014. Lipolysis in Tulum cheese produced from raw and pasteurized goats' milk during ripening. Small Ruminant Research, 121, 351-360.
- Tarakci, Z., Ekici K., Sağdıc, O. and Kücüköner, E., 2005. The effect of black cumin on ripening of Tulum cheese. Archiv für Lebensmittelhygiene, 56, 135-139.
- Tomar, O., Akarca, G., Beykaya, M., Çağlar, A. 2018. Some characteristics of Erzincan Tulum cheese produced using different probiotic cultures and packaging material. Kafkas Veterinary Journal, 24(5), 647-654. DOI: 10.9775/kvfd.2018.19596
- TS-3001, 2016. Turkish standard-tulum cheese. Turkey. ICS 67. 100: 30.
- Turkish Food Codex Regulation–Microbiological Criteria Communique´ (2009/68).