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# THE CHARACTERISTICS OF GOAT SKINS USED IN THE PRODUCTION OF TULUM CHEESE AND CHANGES IN RIPENING ENVIRONMENTS

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# ABSTRACT

The present study aimed to determine the physical, chemical, and textural properties of goatskin used in the production of Tulum cheese and the traditional and industrial ripening conditions. Natural ripening conditions (caves) and some parameters (temperature, humidity, oxygen, and CO<sub>2</sub> values) of the cold storage of the dairy plants were examined between March and October. It was determined that the temperature and moisture content of the natural cave where cheese ripening was carried out was different from that of the dairy factory storage conditions. The oxygen and carbon dioxide ratios were similar in both ripening conditions. The effects of skin thickness, fat, and skin structure, especially tensile strength, elongation break, tear strength, air, water vapor, and oxygen permeability properties were statistically significant. The goatskins' tensile strength, elongation at break and tear strength values were 9.16-36.48 daN, 22.57-81.57 N, and 35.4-150.28 N/mm, respectively. When used as a casing material for the ripening of Tulum cheese, the effect of the skin's properties such as Oxygen, water vapor, and air permeability on the product was found to be significant. The present study determined that goatskin's outward air permeability was 6.6-10.95 times higher than the inward air permeability.

Keywords: Tulum cheese, goatskin, ripening, packaging

# TULUM PEYNİRİ ÜRETİMİNDE KULLANILAN KEÇİ DERİLERİNİN KARAKTERİSTİKLERİ VE OLGUNLAŞTIRMA ORTAMLARINDAKİ DEĞİŞİMLER

# ÖΖ

Çalışma, Tulum peyniri üretiminde kullanılan keçi derisinin fiziksel, kimyasal ve dokusal özelliklerini ve geleneksel olgunlaşma koşullarını belirlemeyi amaçlamaktadır. Çalışmada, Mart ve Ekim ayları arasında doğal olgunlaşma koşulları (mağara) ve süt işletmelerinin soğuk hava depolarının bazı parametreleri (sıcaklık, nem, oksijen ve CO<sub>2</sub> miktarı) incelenmiştir. Peynir olgunlaşmasının gerçekleştirildiği doğal mağara sıcaklık ve nem içeriğinin işletme koşullarına göre daha yüksek olduğu saptanmıştır. Her iki olgunlaşma koşulunda oksijen ve karbondioksit oranı benzer bulunmuştur. Deri kalınlığı, yağ ve deri maddesi içeriği özellikle kopma dayanımı, uzama ve yırtılma, hava, su buharı ve

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İlhan Gün; ORCID no: 0000-0003-0047-273X Zeynep B. Güzel-Seydim; ORCID no: 0000-0002-1536-6545 oksijen geçirgenliği üzerine etkisi istatistiksel olarak önemli bulunmuştur. Derilerin kopma dayanımı, uzama ve yırtılma değerleri sırasıyla 9.16-36.48 daN, 22.57-81.57 N ve 35.4-150.28 N/mm olarak belirlenmiştir. Ayrıca tulum peynirinin olgunlaşması için ambalaj malzemesi olarak kullanıldığında derinin oksijen, su buharı ve hava geçirgenliği gibi özelliklerinin ürün üzerindeki etkisi önemlidir. Bu çalışmada keçi derisinin içten dışa doğru hava geçirgenliğinin, dıştan içe doğru hava geçirgenliğinden 6.6-10.95 kat daha fazla olduğu tespit edilmiştir.

Anahtar kelimeler: Tulum peyniri, keçi derisi, olgunlaşma, ambalaj

# INTRODUCTION

Tulum cheese is one of the most common cheeses consumed in Turkey and differs from other cheese types in terms of its characteristics. Tulum cheese had been previously produced locally on a small scale, however, over time, due to increasing consumer interest, it has been produced in larger quantities and exported. It has been reported to support regional and international development as a branded product among traditional cheeses (Ciftci and Durukan, 2020). Goat skin has been used for cheese ripening and is called Tulum in Turkish. Currently, it is replaced by semi-synthetic salami cases, cloth, plastic, glass, cube, or pottery due to the effect of many factors such as the decrease in the number of small cattle, legal obstacles brought by the forest law, regulations framework established within the of harmonization with the European Union laws, and the ease of use (Duman-Aydın and Gülmez, 2008; Gün et al., 2015; Rençber, 2016; Arslaner and Türkmen, 2020). However, many researchers state that Tulum cheeses obtained from these materials do not provide the desired characteristic of Tulum cheese taste and aroma (Guven and Konar, 1994; Koçak et al., 1996; Tomar et al., 2018). On the other hand, another study emphasized that the ripening of Tulum cheese in glass jars and plastic packaging also positively affected the sensory qualities (Bostan, 1991; Tarakçı et al., 2005).

Goatskin has been used as a packaging material in the production of Tulum cheese for centuries as well as in many areas in the leather industry. It is a unique natural product with its chemical composition and other properties (Toptas, 1998). The skin has many functions in the animal body. The most important ones are protecting the body against external warm/cold conditions, mechanical effects. ultraviolet ravs. and microorganisms, and balancing the body

temperature. Skin is a complex organic substance surrounding the animal body from the outside as a sheath and consists of various tissues formed by various cell units. It is similar in terms of histological and chemical properties, however, it is a unique structure that changes according to each animal species (Orpak, 2001). The use of goatskin as a casing/ripening material in the production of Tulum cheese may vary among different regions. Tulum cheese is generally stuffed into the inner part of the skin. However, the cheese is stuffed on the outer hairy surface after the skin hair is shaved in the Aegean Region, especially in and around the Manisa province. The skin that will be used as the packaging material must be prepared for use after shaving the hair of the goat (Dağ, 2020). Although this is not regarded as essential in production due to the economical circumstances, it creates serious hygiene and economical problems during the supply, production, and storage of the material in industrial production. Also, goatskin is important in terms of durability and protection and should not pose hygienic problems. However, it has been stated that the skin used in the standard production of Tulum cheese should not belong to an animal with anthrax to prevent disease transmission to humans (Anonymous, 2016a). Therefore, the hygienic risk of the skins to be used in Tulum cheese and the challenges during industrial production have focused the research on the use of alternative packaging materials for skin bags. However, the most significant deficiency seen in alternative casing/packaging materials for Tulum cheese is the absence of the structural and permeability properties of the skin. Especially the water vapor permeability of the skin and the permeability of oxygen and carbon dioxide are important criteria. Although the permeability of many alternative materials does not show similar properties to that of goatskin, its permeability is prevented by being coated with

barrier materials. For example, while hygienic glass packaging is an advantage, its lack of permeability is an important disadvantage in cheese ripening. Primarily, in products in which moisture content is not balanced, various defects can be observed including body softening, oversour, and bitter taste (Hayaloglu et al., 2007a).

In the ripening process of Tulum cheese, the characteristics of the ripening environments are as important as the characteristics of the casing/packaging material. The importance of natural cheese ripening environments has been historically well-known. For example, the worldfamous French cheese, Roquefort, is ripened in the Combalou mountain caves. Vertical faults in these caves provide natural ventilation and a unique ripening environment. Caves provide a storage area that keeps a constant temperature of 9°C and humidity of 95% (Renneberg et a., 2017). Thus, the cheese stored in the cave is properly ventilated. A similar case is observed in Kars Kashar cheese ripening rooms. Producers use the northerly winds for good ventilation and ripening of the Kashar cheese and adjust the windows in the direction of the wind (Demir, 2016). Similarly, Tulum cheese is traditionally ripened in natural caves.

The ripening of Tulum cheese in the goatskin under natural conditions provides important textural and flavor characteristics. The previous studies do not provide adequate information on the chemical, physical, and permeability properties of goatskin used in packaging. The present study aimed to determine: 1-Some properties of the goatskins used in the production of traditional Tulum cheese, 2- Conditions of ripening environments (natural caves and storages of cheese plants) between March and October.

## MATERIAL AND METHODS Material

Ten dried goatskins from different manufacturers were obtained from the Burdur province and its districts. Goatskins were preserved by salinating.

# **Ripening Environments**

The data on the cold storage conditions of seven companies that produce Tulum cheese, two caves in the neighboring villages of the Barla District in the Isparta province, and one cave in the village of the Atabey district, where Tulum cheese is ripened in goatskins, were determined. Among these caves, which are 40-80 km away from Isparta, the Kapacık village sinkhole is approximately 25-40-meter deep and has an area of 80-90 m<sup>2</sup>.

# Preparation of Skin Samples for Test and Analysis

The physical and chemical properties of the skins were tested according to TS EN ISO 2418 (Anonymous 2017a) and TS EN ISO 2419 (Anonymous, 2012), respectively. The samples were tested after being kept for 48 hours at 20°C in a standard room with 65% relative humidity (Anonymous, 2012; 2017a). For chemical analysis, the skins were pre-dried at 50°C and used after grinding in a Retsch Mühle (Haan, Germany) cutter mill (Anonymous, 2017b).

# Histopathological Method

Tissue samples were fixed in a 10% buffered formalin solution. The tissue samples were processed using tissue processing equipment (Leica ASP300S, Leica Microsystem, Nussloch, Germany) and embedded in paraffin wax. A fully automatic rotary microtome took five-micron thickness sections from the paraffin blocks (Leica 2155, Leica Microsystem, Nussloch, Germany). After drying, the preparations were passed through alcohol and xylol series, stained with hematoxylin-eosin (HE), mounted with a coverslip, and examined under a light microscope (Luna, 1968).

# Physical Tests of the Samples

The samples were conditioned and prepared following the related standards (Anonymous, 2012; 2017a). The thickness values were measured using a digital micrometer (Satra STD 483, UK) (Anonymous, 2016c). The morphological structures were displayed using a scanning electron microscope (Jeol JSM-6060LV Model, Tokyo, Japan).

#### Chemical Analysis of Skin Samples Sample Preparation for Chemical Analysis

Skin samples were cut into 10-mm-squares according to TS EN ISO 4044 (Anonymous, 2017b). Since raw skins deteriorate due to their protein and fat contents, samples were taken to the pre-drying process in an oven at 50°C. Then, the samples were ground in a Retsch Mühle (Haan, Germany) cutter mill with a 700-1000rpm-rotation speed and a 4-mm-diameter perforated sieve table. The prepared samples were mixed thoroughly and stored in clean, dry, and airtight plastic bags.

#### The pH Value of the Skins

The samples were weighed at  $5\pm0.05g$ , 100 mL of distilled water was added, and shaken for 6 hours by an orbital shaker. Following the filtering, the acidity of the filtrate was measured by a pH meter (Metrohm 827, Sweden) (Anonymous, 2018a).

#### Hide Substance Analysis

After weighing 0.3-0.5 g of the prepared skin samples, the total nitrogen content was analyzed by the Kjeldahl method (Velp UDK 126 D, Italy). The amount of hide substance was calculated by multiplying the percentile nitrogen value by the coefficient of 5.75 (Anonymous, 2018b).

#### **Fat Analysis**

Fat analysis of the skin samples was carried out adopting the Randall Method. The samples were weighed (4 g) and placed in a solvent extraction device (Anonymous, 2018c).

# Tensile Strength and Elongation at Break Test

The tensile strength tests of the samples were performed according to the related standard (TS EN ISO 3376). The specimens were taken from the mid-E point from the mid-point of E, E, and AB, and the mid-point of E and CD, of the skin (Shimadzu AG IS, Japan) (Anonymous, 2016b). The arithmetic mean of the measurements for the thickness of the test samples was taken.

### **Tear Strength Test**

The curved ends of the tool to the skin sample were attached and the device was run (Shimadzu

AG IS, Japan). The value of the force applied to the skin sample at the time of complete tearing was determined. (Anonymous, 2020).

#### Water Vapor Permeability Test

Water vapor permeability tests were performed according to the ASTM F1249-0 standard by Satra STM 473 (UK) (Anonymous, 2011a). The test sample (outer diameter 34 mm) is placed in the mouth of the bottles with freshly-dried silica gel at the rate of 2/3. At the end of the conditioning (16-24 hours), the bottles are removed from the device, and freshly dried silicas brought to a constant weight in the desiccator are placed in the second bottle and the skin samples are placed. The second bottle is weighed as quickly as possible on a precision scale (initial weighing) to record the weighing weight and time. The bottles are placed in the carrier, and the device is operated for 16 hours. Subsequently, the bottles are taken and weighed (final weighing), and the time is recorded.

#### **Oxygen Permeability Analysis**

After being conditioned in accordance with the standard (Anonymous, 2012, 2017a), the air permeability values of the samples were determined in the air permeability measuring device (Devotrans, Istanbul, Turkiye). Measurements were carried out at 23°C, 0% relative humidity, and 1 bar pressure (Anonymous, 2010).

#### Visual Displays of the Samples by Scanning Electron Microscope (SEM)

The samples were taken from the skins. The morphology of the skin's surface and cross-section of the skins were investigated under SEM (Jeol JSM-6060LV, Tokyo, Japan).

#### **Ripening Conditions Analysis**

Temperature and humidity measurements of the natural ripening place and dairy factory were measured by the USB Data Logger (Extech Instruments, RHT10, Waltham, USA). Oxygen and carbon dioxide values were measured using GMI Visa (4-Gas Version, Scotland-England) instruments.

### **Statistical Analysis**

The analysis of variance (ANOVA) was performed using the general linear model (GLM) procedure to compare the different treatments at a 95% confidence level. TUKEY's multiple comparison test performed pairwise comparisons of statistically significant features. The correlation of goatskins variables was estimated by the Pearson correlation coefficient. Statistical analysis of the experimental data was performed using the SPSS 13.0 statistical software.

# Results and Discussion Physical properties of goatskin

In the production of Tulum cheese, goat and sheepskins, called Tulum or Tuluk, are used as a whole. However, there are also Tulum cheeses that are offered for sale in smaller skins of 3-5 kg (Ozturkoglu-Budak et al., 2015). In the study, goat skin was purchased as a whole, which had no scratches or tears and was used in the analysis. The size of the skins used s used in the present study was approximately 0.6-0.7 m<sup>2</sup>. Gerhard (1996) has stated that the size of goatskin is approximately 0.5-0.9 m<sup>2</sup>, and that of suckling lamb is 0.2-0.5 m<sup>2</sup>. There must be no deformations such as scratches and tears on the goatskin surface to prevent Tulum Cheese quality. Deformations such as cuts or tears on the skin can accelerate the deterioration of the cheese towards the interior by bacterial contamination or contact with air. Assefa et al. (2012) found that a scratch was detected in 22.5%, and a scar was detected in 11.3% of 400 skin samples. These skins must be sewn in such a way so that they don't let air pass through before curd pressing into the Tulum.

In the present, differences were determined between the thickness values of the samples (Table 1). This was mainly associated with the animal's age or body weight (Thiruvenkadan and Panneerselvam, 2008). It has been reported that the goatskin thickness of a 5-10 kg body weight was 2.44 mm, while the goatskin thickness of a 25 kg body weight was 3.37 mm. The dermis layer, known as the chorium or cutis, is the part of the animal body that protects against physical impacts/hits and injury, balances the body temperature, and constitutes approximately 85% of the total skin thickness (Toptaş, 1998). The dermis layer of a male goat consists of thick and dense connective tissue. Therefore, it is indicated that the dermis layer of the thoracal regio of males is thicker than that of females. The epidermis layer, which covers the upper part of the skin, constitutes 1-2% of the total skin thickness (Toptaş, 1998). Furthermore, the papilare and reticulare layers have a smooth collagen fiber distributed irregularly and a solid rough collagen layer (Suwiti et al., 2019). These structures may affect the thickness of the skin.

Goatskins are stronger than sheepskins, and both of them possess air and oxygen permeability (Hayaloglu et al., 2007b). The amount of sebaceous and sweat glands in goatskin is lower and smaller compared to that of sheepskin. Therefore, goatskin is more durable because it does not have а spongy-like structure (Harmancıoğlu and Dikmelik, 1993). The present study showed that goatskin's outward air was 6.6-10.95 times higher than inward air permeability. The results in Table 2 show a positive correlation between goatskins' air and oxygen permeability but a negative correlation between water vapor permeability. In general, the skins of female animals are thinner but more flexible than the skins of males. Since the structural properties of the tissues can be different in young and old animal skins, the rate of substance transfer such as water and air from the skin also changes. Therefore, the amount of water they contain, the protein structure, and the amount and quality of substances differ significantly intercellular (Anonymous, 2011b). The epidermis varies between 1-2 and 6% of the total skin thickness in goatskins. Collagen fiber bundles are thicker and contain more fibers, making goat skins more durable (Orpak, 2001). For all these reasons, it has been thought that the permeability properties such as air and water vapor of the goatskin change.

Skin Properties	1	2	3	4	5	6	7	8	9	10	Mean
-	6.18±	6.56±	5.78±	6.05±	5.80±	6.10±	6.71±	5.80±	5.85±	6.61±	6.15±
рН	0.03 <sup>c2</sup>	0.01 <sup>b</sup>	0.02e	0.02 <sup>d</sup>	0.02 <sup>e</sup>	0.01 <sup>cd</sup>	0.01 <sup>ab</sup>	0.03e	0.04e	0.03ª	0.03
$\mathbf{E} \in \langle 0/ \rangle$	1.38±	10.21±	4.28±	14.56±	2.24±	$2.07 \pm$	5.76±	3.80±	6.16±	7.16±	5.76±
Fat (%)	0.03 <sup>i</sup>	0.03b	$0.02^{f}$	0.32 <sup>a</sup>	0.06 <sup>h</sup>	0.11 <sup>h</sup>	0.02e	0.09g	0.03 <sup>d</sup>	0.02c	0.15
Total nitroion (0/)	10.77	15.84±	15.98±	16.85±	14.28	13.44	14.61	12.81	14.81	16.48	15.01±
Total nitrojen (%)	$\pm 0.05^{i}$	0.03c	0.03c	0.20ª	$\pm 0.04^{\rm f}$	$\pm 0.05$ g	$\pm 0.02^{\circ}$	$\pm 0.02^{h}$	$\pm 0.02^{d}$	$\pm 0.02^{\text{b}}$	0.18
Ilido Substanzo (9/)	61.95	91.10±	91.87±	96.86±	82.15	77.28	84.05	73.70	85.18	94.78	86.32±
Hide Substance (%)	$\pm 0.22^{i}$	0.19c	0.20c	0.58ª	$\pm 0.23^{\rm f}$	$\pm 0.28$ g	$\pm 0.11^{\circ}$	$\pm 0.12^{h}$	$\pm 0.10^{d}$	$\pm 0.12^{b}$	1.64
Thiskness (mm)	0.54±	0.88±	$0.88 \pm$	1.41±	0.56±	0.63±	0.68±	0.74±	0.83±	0.84±	$0.80 \pm$
Thickness (mm)	0.01d	0.01 <sup>b</sup>	0.02 <sup>b</sup>	0.02a	0.01 <sup>d</sup>	0.01 <sup>d</sup>	0.03 <sup>cd</sup>	0.01c	0.02 <sup>b</sup>	0.01 <sup>b</sup>	0.01
Tensile strength (daN)	9.16±	36.48±	15.38±	25.68±	15.79	14.42	18.28	18.31	19.42	28.71	20.17±
Tensile strength (dain)	0.34 <sup>i</sup>	1.20ª	0.02g	0.02c	$\pm 0.03^{\rm f}$	$\pm 0.11^{h}$	$\pm 0.03^{\circ}$	$\pm 0.03^{\rm e}$	$\pm 0.06^{d}$	$\pm 0.18^{\text{b}}$	2.69
Elemention at Proals (0/)	22.57	65.37±	55.79±	81.57±	25.37	$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	57.78	47.80±			
Elongation at Break (%)	$\pm 0.19^{i}$	0.08b	0.09e	2.20ª	$\pm 0.06^{h}$	$\pm 0.23^{h}$	$\pm 0.04^{\text{f}}$	$\pm 0.03^{\text{g}}$	$\pm 0.06^{\circ}$	$\pm 0.04^{d}$	7.99
Tear strength (N/mm)	35.40	119.91	110.94	150.28	42.07	44.23	78.47	80.19	95.54	88.36	84.26±
rear strength (iv/min)	$\pm 3.20^{h}$	±0.39b	$\pm 0.06^{\circ}$	$\pm 4.24^{a}$	$\pm 0.04$ g	$\pm 0.06$ g	$\pm 0.25^{\text{f}}$	$\pm 0.09^{\text{f}}$	$\pm 0.24^{d}$	$\pm 0.06^{\circ}$	11.88
Air Permeability	12670	883±	618±	400±	8393±	7648±	6493±	$1232\pm$	1308±	1318±	3760
(from outside to inside	$\pm 6.25^{a}$	49f	25 <sup>f</sup>	400 <u>+</u> 2.67g	11 <sup>b</sup>	42°	26 <sup>d</sup>	12 <u>52</u> 27e	1308 <u>+</u> 12e	21°	$\pm 3.15$
surface) (Liter/day)	±0.25"	т <i>у</i> "	25	2.078	11.	741	20"	21	12	21	±5.15
Air Permeability	83520	4380±	8551±	4544±	45425	37112	14746	28964	17644	16894	26130
(from inside to outside	$\pm 20.4^{a}$	5.32 <sup>k</sup>	65 <sup>i</sup>	41 <sup>k</sup>	±116 <sup>b</sup>	$\pm 184^{\circ}$	$\pm 30^{h}$	$\pm 20004$	$\pm 112^{\circ}$	$\pm 86^{\text{fg}}$	$\pm 23.78$
surface) (Liter/day)	-20.1	5.52	05	11	2110	2101	250	1200	±112	2000	-23.70
Water Vapor	3320.6	1511±	2687±	1234.9	1670±	2263±	2629±	3128±	$2969 \pm$	$2870 \pm$	2423.3
Permeability (mg	$\pm 32^{a}$	7g	9de	±8.26 <sup>h</sup>	23g	71 <sup>f</sup>	43°	21 <sup>ab</sup>	38 <sup>bc</sup>	42 <sup>cd</sup>	$\pm 535$
H <sub>2</sub> O/cm <sup>2</sup> h)			-						••	.=	
Oxygen Permeability	4481±	619±2.	574±5.	229.1±	2310±	1667±	1603±	1484±	1370±	1089±	1538.21
$(cc/m^2 day)$	26.84ª	52g	87g	3.42 <sup>h</sup>	29.1 <sup>b</sup>	14.5°	21.7c	15.2 <sup>d</sup>	8.65 <sup>e</sup>	8.22 <sup>f</sup>	$\pm 820$

Table 1. Some chemical and physical properties of skin samples<sup>1</sup>

<sup>1</sup> Data are the averages of triplicates <sup>2 a, b</sup>: Means within a row with different lowercase letters show significant differences between goatskin samples, P < 0.05

Table 2. Correlation of the skin properties

Variables		pН	Fat	Total Nitrojen	Hide Substance	Thickness	Tensile Strength	Elongation at Break	Tear Strength	Air Permeability <sup>a</sup>	Air Permeability <sup>b</sup>	Water Vapor Permeability	Oxygen Permability
	Pearson Correlation	1	.301	.213	.213	013	.507	.145	.088	.057	192		049
рН	Sig. (2-tailed)		.398	.554	.554	.971	.135	.690	.809	.875	.596	.855	.892
	Ν	10	10	10	10	10	10	10	10	10	10	10	10
E-4	Pearson Correlation	.301	1	.750*	.750*	.916**	.779**	.918**	.898**	671*	720*	592	700*
Fat	Sig. (2-tailed)	.398		.013	.013	.000	.008	.000	.000	.034	.019	.071	.024
	Ν	10	10	10	10	10	10	10	10	10	10	10	10
Total	Pearson Correlation	.213	.750*	1	1.000**	.718*	.712*	.819**	.794**	761*	887**	531	883**
Nitrojen	Sig. (2-tailed)	.554	.013		.000	.019	.021	.004	.006	.011	.001	.115	.001
	Ν	10	10	10	10	10	10	10	10	10	10	10	10
Hide	Pearson Correlation	.213	.750*	1.000**	1	.718*	.712*	.819**	.794**	761*	886**	530	883**
Substance	Sig. (2-tailed)	.554	.013	.000		.019	.021	.004	.006	.011	.001	.115	.001
	Ν	10	10	10	10	10	10	10	10	10	10	10	10
	Pearson Correlation	013	.916**	.718*	.718*	1	.552	.908**	.914**	717*	677*	494	720*
Thickness	Sig. (2-tailed)	.971	.000	.019	.019		.098	.000	.000	.020	.031	.146	.019
	Ν	10	10	10	10	10	10	10	10	10	10	10	10
Tensile	Pearson Correlation	.507	.779**	.712*	.712*	.552	1	.732*	.688*	673*	700*	500	670*
Strength	Sig. (2-tailed)	.135	.008	.021	.021	.098		.016	.028	.033	.024	.141	.034
	Ν	10	10	10	10	10	10	10	10	10	10	10	10
Elongation	Pearson nCorrelation	.145	.918**	.819**	.819**	.908**	.732*	1	.973**	857**	821**	369	793**
	Sig. (2-tailed)	.690	.000	.004	.004	.000	.016		.000	.002	.004	.294	.006
	Ν	10	10	10	10	10	10	10	10	10	10	10	10

			-		00	auton o		min prop	Jeruco	(commu	-)		
Variables		pН	Fat	Total Nitrojen	Hide Substance	Thickness	Tensile Strength	Elongation at Break	Tear Strength	Air Permeability <sup>a</sup>		Water Vapor Permeability	
Tear	Pearson Correlation	.088	.898**	.794**	.794**	.914**	.688*	.973**	1	865**	847**	413	832**
Strength	Sig. (2-tailed)	.809	.000	.006	.006	.000	.028	.000		.001	.002	.236	.003
_	N	10	10	10	10	10	10	10	10	10	10	10	10
Air	Pearson Correlation	.057	671*	761*	761*	717*	673*	857**	865**	1	.874**	.177	.891**
Permeability	<sup>a</sup> Sig. (2-tailed)	.875	.034	.011	.011	.020	.033	.002	.001		.001	.624	.001
	N	10	10	10	10	10	10	10	10	10	10	10	10
Air	Pearson Correlation	192	720*	887**	886**	677*	700*	821**	847**	.874**	1	.395	.969**
Permeability	<sup>b</sup> Sig. (2-tailed)	.596	.019	.001	.001	.031	.024	.004	.002	.001		.259	.000
	N	10	10	10	10	10	10	10	10	10	10	10	10
Water Vapo	Pearson Correlation	067	592	531	530	494	500	369	413	.177	.395	1	.465
	Sig. (2-tailed)	.855	.071	.115	.115	.146	.141	.294	.236	.624	.259		.176
	N	10	10	10	10	10	10	10	10	10	10	10	10
Oxygen	Pearson Correlation	049	700*	883**	883**	720*	670*	793**	832**	.891**	.969**	.465	1
	Sig. (2-tailed)	.892	.024	.001	.001	.019	.034	.006	.003	.001	.000	.176	
	N	10	10	10	10	10	10	10	10	10	10	10	10

Table 2. Correlation of the skin properties (continue)

\*Correlation is significant at the 0.05 level (2-tailed)

\*\*Correlation is significant at the 0.01 level (2-tailed)

<sup>a</sup>From outside to inside surface (Liter/day)

<sup>b</sup>From inside to outside surface (Liter/day)

Similarly, tensile strength, elongation at break, and tear strength values of the skin samples showed differences. These three parameters provide important information about the strong structure of the skin. According to the findings detected in different gender and age groups, the tensile strength and elongation at break values of hair goatskins were 12.3-18.0 N/mm<sup>2</sup> and 38.16-65.92%, respectively (Gün et al., 2015). The tear strength values of the skin samples were between 34.75 and 56.29 N/mm, and the water vapor and air permeability values were between 0.46 and 0.82 mg/cm<sup>2</sup>.h, and 9.27 and 38.90 cm<sup>3</sup>/cm<sup>2</sup>.sec, respectively. In a study on Sudan desert goatskin, elongation at break, tensile strength, and flexibility degree values were 58.3%, 194.4 kg/cm<sup>2</sup>, and 3.3 in the male goatskin, respectively. However, the same values were 61.8%, 159.1 kg/cm<sup>2</sup>, and 2.1 in female goats, respectively (Ebrahiem, 2016). These physical properties were associated with the non-homogeneous distribution of the collagen structure in the skin (Maxwell, 2007). This shows that it stretches better when the skin has a higher elongation value, and there can be more space between the fibers for moisture loss. Therefore, it is easier to remove the water from the cheese, and, hence, the cheese can be preserved for a longer time. Thus, the chemical,

biochemical, and microbiological quality of the cheese would slightly change.

A histopathological examination was carried out to determine a possible deterioration in the skin tissue, and the results are given in Figure 1. Accordingly, no lesions were observed in the skin samples. The tissue sample of an unhealthy goatskin is shown in Figure 2. The epidermis, one of the skin layers, consists of epithelial cells, including hair follicles and sweat and sebaceous glands, and is located in the skin (Figure 1). All layers of the skin are included in Tulum cheese however, the epidermis is removed from the original skin layer during the tanning process in the leather industry.

#### Chemical properties of goatskin

The chemical analysis results of the goatskins used in this study are presented in Table 1. The pH values in skin samples were between 5.78 and 6.71. Since anionic or cationic values can be obtained according to the amino acid content of the skin, pH values can vary (Harmancioglu and Dikmelik, 1993). Gün et al. (2015) determined that the pH values of the skin samples changed in the range of 6.3-6.6. However, in the leather industry, the pH value changes due to tanning (Nasr et al., 2013). The fat in goatskin prepared for cheese pressing is affected by the fat in the skin structure and the amount of residual fat in the skin that has not been adequately washed. Mainly meat and adipose tissue residues can remain on the skin during the salting and drying phase and change the skin properties. It was determined that the fat and hide substance content of the skin samples varied significantly between 1.38 and 14.56%, and 61.95 and 96.86%, respectively. It was observed that the fat value varies considerably in goat samples of different breeds. The fat value was determined to be 5% (Dikmelik, 1978), 2.77-8.08% (Gün et al., 2015) in hair goatskin and 11% (Eke, 1997) in angora goatskin, and 30% (Sharphause, 1983) in sheepskin. Lipolysis and proteolysis of fat and hide nitrogen content in the skin structure can take place by the microorganisms found on the

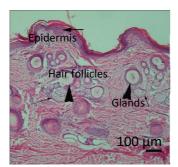


Figure 1. Textural structure of goatskin

Furthermore, an increase in the composition of fat and hide substance in the skin significantly affects the physical properties of the skin (Table 2). According to the correlation of the skin variables, it was found that an increase in the fat and hide substance ratio has positive effects on thickness, tensile strength, elongation at break and tear strength, and adverse effects on air permeability, water vapor permeability, and oxygen permeability properties of the goatskin. In other words, the increase in skin thickness, fat, and protein content significantly reduces air, water vapor, and oxygen permeability.

In the present study, the hide substance value belonging to hair goats was 86.32%. The correlation between the hide substance and tensile strength variables was statistically significant as

skin or the raw cheese curd. Therefore, the bitter peptides that occur can cause the cheese to exert a rancid aroma, especially on the inner surface of the skin. Mainly meat and adipose tissue residues can remain on the skin during the salting and drying phase and can change the skin properties. The hypodermis, the lower skin layer, consists of connective tissue that provides the bond between the skin and the animal's body. This layer consists of loose connective tissues and a fat layer (Suwiti et al., 2019). Since this layer is not removed properly, it is thought that the fat content, air, oxygen, and water vapor permeability of the goatskin to be used as cheese packaging material will be affected. In addition, it is thought that it will affect many parameters such as moisture loss, texture, and aroma compounds formation during cheese ripening.

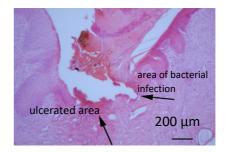


Figure 2. Textural structure of ulcer-infected goatskin

the case in tear strength and elongation at break (P < 0.05) (Table 2). Previous studies determined that the hide substance value of hair goats was 44-64% (Eke, 1997) and 81.72% (Dikmelik, 1978). In two-year-old animals, this value was determined to be 80.28% in females and 77.30% in males (Eke, 1997). In another study on hair goatskins properties, the hide substance ratio was 51.81-62.89% (Gün et al., 2015). The protein structure of the skin is composed of collagen, which has a fibrillar structure. During moistening of the dried skin, the skin becomes flexible by expanding the volume of collagen with the swelling property. This is related to water particles entering the collagen fibrils with osmotic pressure (Harmancioglu and Dikmelik, 1993). Since this structure forms the basis of the skin and provides the skin with its physical properties, it is described as a hide substance (Eke, 1997). Depending on the animal species, the changes in the collagen value also affect the characteristics of Tulum cheese pressed in these goatskin casings (Gün et al., 2015). It is thought that the increase in hide substance and fat content may also affect the oxygen and water vapor permeability of the skin. According to the correlation of the skin variables, an increase in fat and hide substance ratio has a significant positive effect on thickness, tensile strength, elongation at break and tear strength, whereas a negative effect on air permeability, water vapor permeability, and oxygen permeability properties of the goatskin. In other words, the increase in skin thickness, fat, and protein content significantly reduces air, water vapor, and oxygen permeability.

### SEM Images of Goatskin

# Visual Displays of the Samples by Scanning Electron Microscope.

Photographs of goat skins taken using a scanning electron microscope are shown in Figure 3.

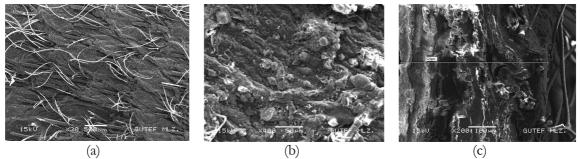


Figure 3. SEM images of the outer surface (a, 30 times magnification), the inner surface (b, 400 times magnification), and the vertical orientation (c, 200 times magnification) of skin samples

The cubic crystal structures of the sodium chloride molecules can be seen on the skin's surface by SEM micrograph. The sizes of salt crystals were between 10 and 20 micrometers. The average pore size of the skins was found to be 15 micrometers. It was thought that the small size of the salt molecules facilitated its penetration into the cross-section of the skin, and thus the skins were preserved by salt molecules effectively. Despite the cleaning of the skin to be used in the production of Tulum cheese, photographs showed that the hairs and other residues on the outer surface remained. As seen in the scanning electron microscope images, the fat meat residues on the inner surfaces and the hairs on the outer surface affect the skin's water vapor and gas permeability, thickness, and strength (Table 2). During cheese ripening, microorganisms, especially yeast and mold, play an important role. Since the whey released from the cheese curd and the moisture absorbed from the environment causes the skin's outer surface to remain moist, it is thought that unwanted microorganism growth can take place.

#### A Natural Cave and Cold Storage Conditions for the Ripening of Tulum Cheese

In the present study, data were collected every 15 days during the 8-month (March-October) ripening period in which traditional production is carried out. Therefore, data collection was initiated and ended simultaneously with the ripening periods in natural cave conditions and cold storage in dairy factories. Data on the cold storage conditions of seven companies that produce Tulum cheese, two caves in the neighboring villages of the Barla District of Isparta province, and one cave (called sinkhole) in the village of Atabey district, where Tulum cheese is ripened in goatskin, were collected (Tables 3 and 4). Among these caves, which are 40-80 km away from Isparta, the Kapacık village sinkhole is approximately 25-40 m deep and has an area of 80-90 m<sup>2</sup> (Figure 4).

The average temperature in the caves was 7.03°C, and the relative humidity was 92.30%. The atmospheric composition consisted of 20.91% oxygen, however, it was determined that the

carbon dioxide levels were below the limit value that the device use could read. The mean storage temperature and the relative humidities were 5.37°C and 77.65%, respectively, in the cold storage of the Tulum cheese companies. Due to the airflow in cheese ripening rooms and caves, it was deemed that the environment had an appropriate atmospheric composition. No information was found in the literature regarding such an evaluation. Similarly, caves are used in the ripening of other international cheeses. For example, the ripening process of Roquefort cheese is traditionally carried out in the mountain caves of Combalou. The natural ventilation of the cave is due to the formation of the cave by vertical faults providing airflow. Thus, a natural storage area is provided in the caves, keeping the temperature constant at 9°C and humidity at 95%. (Renneberg et a., 2017). Taga cheese's ripening, traditional Romanian cheese, is also carried out in the cave with a unique microflora. This cave has a temperature of 14-15°C and 90-95% humidity (Criste et al., 2020). The one-year average temperature and humidity of the Peynirini Cave were found to be 6°C and 99.6%, respectively (Dağlı et al., 2020).

Many world-famous cheese types are ripened either in caves or in special cellars (Tudor Kalit et al., 2010, Kalit et al., 2016). Especially, cave conditions should be suitable for the quality of the cheese produced. Caves are used to ripen skin Tulum cheese, especially in the highlands of Eastern Anatolia, Central Anatolia, Eastern Black Sea, and Mediterranean regions in Turkey. The internal temperature of the Divle sinkhole is 5-10°C, and the relative humidity is 85% (Kan et al., 2010; Ozturkoglu-Budak et al., 2015). In the Geographical Indication Registry Document (CIRC), it is emphasized that the temperature of the Divle sinkhole should be +4°C and the humidity should be 80% (Anonymous, 2017c). However, the temperature of many plateaus in summer can vary in the range of 12-18°C, depending on the altitude. Day and night temperatures can be different in these regions (Kirdar et al., 2015). It is stated that the unique cellars where Erzincan Savak Tulum cheese is ripened feature 6-8°C temperature and 75-80%

relative humidity (Akyüz, 1981). However, it has been stated in the CIRC that Erzincan Tulum cheese is matured for 4 months at  $-2 - 0^{\circ}$ C (Anonymous, 2001).

During this study, it was observed that Tulum cheese is stored in plastic bags at 20-25°C for 5-7 days before the curd is produced in industrial production and then ripened at low storage temperatures by pressing the skin. In addition, it was determined that the cold storage of the dairy factory is not only used for Tulum cheese but also for other dairy products, therefore, the temperature of the cold storage is kept lower. According to the data obtained in the present study, it was determined that only 2 of 7 different Tulum cheese plants producing Tulum cheese kept the products at an average of 2°C. The mean temperature of the dairy factory producing only Tulum cheese was determined to be 7-9°C. This temperature almost was the same as that of the cave conditions. It was observed that, apart from the temperature data of the ripening room belonging to two dairy plants, the temperature data of the other companies were similar to the data obtained in the caves. Kurt and Öztek (1984) determined the storage temperature of the Erzincan Meat and Fish Institution, where Tulum cheeses are stored, to be 2-3°C and relative humidity to be 80-82%. The findings obtained in the present study were compatible with the data reported by Kurt and Öztek (1984). The high temperature and moisture content of the natural ripening conditions significantly affect the cheese microflora and enzymatic activity, thus changing the chemical, biochemical and microbiological quality of the Tulum cheese (Tekin and Güler, 2019). Also, the growth of yeast and mold on the goatskin under natural ripening conditions and the reproduction of insect species such as mites on the skin and fecal residues of these insects can pose a risk in terms of food safety. It has been stated that biogenic amines formed as a result of microbial decarboxylation from amino acids as a result of proteolysis during ripening increase in cheese samples. Especially, histamine, tyramine, tryptamine putrescine, and cadaverine levels increase (Durlu-Özkaya et al., 2000). In Turkey, there are no standards on production technology

in the production of Tulum cheese. The ripening conditions of cheeses produced, especially in the highlands and for those stored in natural environments vary. Therefore, the characteristics of Tulum cheeses vary. Standardizing the industrial production conditions will eliminate the risks in the production and storage of the Tulum cheese. Even if the production standardization of the products that have received the CI certificate has been achieved, there are currently no standards regarding the skins used. In particular, the properties of goat/sheep skins used in the cheese ripening with CI certificates should be examined.



Figure 4. Sinkhole of Kapacık Village

Table 3. Some chara	atomistics of mater	al min anima a	anditiona (as	and similar of	bre months
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Parameter	Parameter			May	June	July	August	September	October
Temperature (	°C)	9.57	8.77	7.40	6.77	6.43	5.83	6.30	6.45
Humidity (%)		86.60	90.27	92.28	92.97	92.42	93.08	94.20	93.73
A turn a sur la sur	Oxygen (%)	20.9	20.9	20.9	20.9	20.9	20.9	20.9	20.9
Atmosphere composition	Carbon dioxide (%)	BLV	BLV	BLV	BLV	BLV	BLV	6.30 94.20	BLV
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BLV: Below the limit value that the device can read

Parameter	March	April	May	June	July	August	September	October	
Temperature (	Temperature (°C)		5.74	5.72	5.33	5.08	5.26	5.27	5.19
Humidity (%)		79.90	80.76	76.94	77.56	76.95	75.91	76.60	77.69
A. 1	Oxygen (%)	20.91	20.96	20.91	20.9	20.9	20.9	20.9	20.9
Atmosphere composition	Carbon dioxide (%)	BLV	BLV	BLV	BLV	BLV	BLV	76.60 20.9	BLV

Table 4. Some characteristics of cold storage conditions in the dairy factory by months

BLV: Below the limit value that the device can read

## CONCLUSION

Tulum cheese is an important type of cheese for Turkey. It is traditionally produced in the highlands during spring and summer. The ripening process following the stuffing/pressing the curd in goatskin is a crucial factor for Tulum cheese to gain its unique taste and aroma. The use of female or male animal skins as a packaging material, in addition to the racial characteristics, can change the quality of Tulum cheese. However, examining the characteristics of goatskin, the chemical, physical and textural properties significantly vary. Also, the change in temperature and humidity values of ripening warehouses is an essential factor that would affect the ripening level of Tulum cheese. Therefore, to obtain a standard quality Tulum cheese, besides the values at the production stages, optimum casing type and storage conditions should be determined and advised. There is no information about the characteristics of traditional ripening materials in products that have received a geographical indication registration certificate. Therefore, it is important to examine the packaging material properties of cheese varieties that have received this certificate and are ripened in the skin.

## **CONFLICT OF INTEREST**

The authors have declared no conflicts of interest for this article.

# **AUTHOR CONTRIBUTIONS**

İlhan Gün and Zeynep B. Güzel-Seydim contributed to the conception and planning of the research. İlhan Gün obtained data from caves and dairy factories. The goat skin analyzes were carried out by İlhan GÜN. Both authors contributed to the writing of the article.

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