# SEASONAL CHANGES IN QUALITY OF HALLOUMI CHEESE PRODUCED FROM COW MILK

# A THESIS SUBMITTED TO THE GRADUATE SCHOOL OF APPLIED SCIENCES OF NEAR EAST UNIVERSITY

By ASLIHAN ESENDAĞLI

In Partial Fullfillment of the Reqirements for the Degree of Master of Science in Food Engineering

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# Ashhan ESENDAĞLI: SEASONAL CHANGES IN QUALITY OF HALLOUMI CHEESE PRODUCED FROM COW MILK

Approval of the Graduate School of Applied Science

### Prof. Dr. Nadire ÇAVUŞ

## We certify this thesis is satisfactory for the award of the degree of Master of Science in Food Engineering

**Examining Committee in Charge:** 

Assist. Prof. Dr. Serdar Susever

Committee Chairman of the Jury, Faculty of Health Sciences, NEU

Dr. Kehinde Adewafe Adesina

Department of Food Engineering, NEU

Assist. Prof. Dr. Perihan Adun

Supervisor, Department of Food Engineering, NEU I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not orginal to this work.

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#### ABSTRACT

The aim of this study was to investigate the effect of seasonal changes on composition of milk and halloumi cheese and also on sensory attributes of halloumi cheese. Changes in fat, dry matter, pH and sensory attributes ( in halloumi cheese only) of these products were analysed during the production period of one year. Monthly weather temperature and humidity forecast data were also collected. It was found that the composition of milk and halloumi cheeses were affected by these seasonal changes in composition. In winter when total dry matter and fat content were high, sensory quality of halloumi cheeses had higher values. pH value of halloumi cheese were stable during these production period.

Keywords: halloumi cheese; milk; seasonal changes; chemical changes; quality

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22-01-19		81
23-01-19		81
24-01-19	11	80
25-01-19		77
26-01-19		83
27-01-19		56
28-01-19		80
29-01-19		77
30-01-19		81
31-01-19		78



# METEOROLOJİ DAİRESİ

ERCAN HAFTALIK TAHMİN RAPORU Tel: (0392) 2314658 Fax: (0392) 2314763 e- mail: <u>ercanmeteor@gmail.com</u>

SIRA NO : ECN - LCEN- 2019/02/07

TARİH : ... 07 – ŞUBAT -- 2019...

08 - 14 - ŞUBAT - 2019 tarihleri arasında bölgemizde beklenen hava durumu :

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10 - ŞUBAT (PZ.): Parçalı bulutlu yer yer sağanak yağmurlu,

11 - ŞUBAT (PT.) : Parçalı az bulutlu,

12 - ŞUBAT (SA.): Az bulutlu öğleden sonra Parçah bulutlu,

13 - ŞUBAT (ÇA.): Parçalı bulutlu öğleden sonra yer yer sağanak yağmurlu,

14 - ŞUBAT (PŞ.): Parçalı çok bulutlu yer yer sağanak yağmurlu , geçmesi beklenmektedir.

En yüksek hava sıcaklığı : Genellikle İç kesimlerde ve Sahillerde 14 – 17 °C seyretmesi beklenmektedir.

Rüzgar : Genllikle KUZEY ve BATI yönlerden orta kuvvette, zaman zaman Kuvvetli , esmesi beklenmektedir.

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ERCAN ORTALAMA SICAKLIK VE NEM DEĞERLERİ

TARİH :	SICAKLIK <sup>Q</sup> C	% NEM
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21-03-18	17	57
22-03-18	16	68
23-03-18		60
24-03-18		55
25-03-18		61
26-03-18		42
27-03-18		42
28-03-18		60
29-03-18		66
30-03-18	15	63
31-03-18	16	63

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# ERCAN ORTALAMA SICAKLIK VE NEM DEĞERLERİ

TARİH :	SICAKLIK ºC	% NEM
20-05-18	30	
21-05-18	29	35
22-05-18	28	49
23-05-18	27	
24-05-18	28	42
25-05-18	25	63
26-05-18	24	58
27-05-18	24	60
28-05-18	25	59
29-05-18	25	63
30-05-18	23	70
31-05-18	22	



# METEOROLOJİ DAİRESİ

ERCAN HAFTALIK TAHMİN RAPORU Tel: (0392) 2314658 Fax: (0392) 2314763 e-mail: <u>ercanmeteor@gmail.com</u>

## SIRA NO : ECN - LCEN- 2018/05/31

TARİH : ... 31 - MAYIS-- 2018...

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Bölgemizin genellikle Alçak basınç sistemi ile Üst atmosferdeki serin ve nemli hava kütlesinin etkisi altında kalması beklenmektedir.

Bölgemizdeki meteorolojik durum nedeniyle hava ;

01 HAZİRAN (CU.): Parçalı bulutlu, yer yer sağanak veya gökgürültülü sağanak yağmurlu,
02 HAZİRAN (CT.): Parçalı bulutlu, yer yer sağanak veya gökgürültülü sağanak yağmurlu,
03 HAZİRAN (PZ.): Parçalı bulutlu, yer yer sağanak veya gökgürültülü sağanak yağmurlu,
04 HAZİRAN (PT.): Parçalı bulutlu, yer yer sağanak veya gökgürültülü sağanak yağmurlu,
05 HAZİRAN (SA.): Az bulutlu, öğleden sonra parçalı bulutlu, yer yer sağanak yağmurlu,
06 HAZİRAN (ÇA.): Az bulutlu, öğleden sonra parçalı bulutlu, yer yer hafif sağanak yağmurlu,
07 HAZİRAN (PŞ.): Açık az bulutlu, geçmesi beklenmektedir.

En yüksek hava sıcaklığı: Genellikle İç kesimlerde 28 - 32 °C ve, Sahillerde 25 - 28 °C, dolaylarında seyretmesi heklenmektedir.

Rüzgar : Genellikle GÜNEY ve BATI yönlerden orta kuvvette , zaman zaman kuvvetli esmesi beklenmektedir.

Meteoroloji Dairesi Müdürlüğü Ercan Meteoroloji İst. CAN VURGUN

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# ERCAN ORTALAMA SICAKLIK VE NEM DEĞERLERİ

TARİH :	SICAKLIK <sup>o</sup> C	% NEM
20-08-18	29	64
21-08-18	29	62
22-08-18	29	60
23-08-18	31	52
24-08-18		60
25-08-18	29	61
26-08-18		
27-08-18		61
28-08-18		60
29-08-18		62
30-08-18		58
31-08-18		73

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#### **CHAPTER 1**

#### **INTRODUCTION**

People first learned to consume milk from other mammals on a regular basis after the domestication of animals during the Neolithic Revolution. This development occurred in Mesopotamia. People first domesticated the most important dairy animals cattle, sheep and goats in Southwest Asia. Domestic dairy animals spread from Asia to Europe (starts around 7000 BC, but did not reach until 4000 BC Britain and Scandinavia) and South Asia (7000-5500 BC). The first farmers in Central Europe and the United Kingdom milked their animals.

Milk is an emulsion of butterfat globules within a water-based fluid that contains dissolved carbohydrates and protein aggregates with minerals. Because it is produced as a food source. It is possible to collect the substances in the structure of milk under five main headings: milk sugar (lactose), milk fat, milk proteins, mineral substances and vitamins. Many of these nutrients in the structure of milk (lactose, casein etc.) are not found in any food rather than milk. 1 liter of milk contains average 36 grams of milk fat, 38 grams of protein, 52 grams of carbohydrates, 7 grams of mineral substances and water and oil-soluble vitamins (ASÜD, 2019; MEGEP, 2016).

Dairy products are produced from milk, such as UHT milk, cream, butter, yoghurt, ice cream, cheese and some others.

According to Turkish Food Codex, pasteurization is used to kill harmful pathogenic bacteria by heating the milk for a short time and then immediately cooling it. The standard high temperature short time (HTST) process of 72 °C for 15 seconds completely kills pathogenic bacteria in milk.

Cheese is another product made from milk. Whole milk is reacted to form curds that can be compressed, processed and stored to form cheese. The cheese milk is pre-treated, possibly pre-ripened after addition of a bacteria culture appropriate to the type of cheese, and mixed with rennet. The enzyme activity of the rennet causes the milk to coagulate into a solid gel known as coagulum. This is cut with special cutting tools into small cubes of the desired size - primarily to facilitate expulsion of whey. During the rest of the curdmaking process, the bacteria grow and multiply and form lactic acid from the lactose. The curd grains are subjected to mechanical treatment with stirring tools, while at the same time the curd is heated, according to a pre-set program. The combined effect of these three actions – growth of bacteria, mechanical treatment and heat treatment - results in syneresis, i.e. expulsion of whey from the curd grains. The finished curd is placed in cheese moulds, mostly made of plastic, which determine the shape and size of the finished cheese. The cheese is pressed, either by its own weight or more commonly by applying pressure to the moulds. Treatment during curdmaking, pressing, brining and storage conditions determines the characteristics of the cheese (Teblig, 2015).

Halloumi is the traditional product of the island of Cyprus and is characterized as a semihard, un-ripened and salty cheese made from a mixture of cow, goat and sheep milk. The Halloumi cheese is produced only by the coagulation of milk with rennet without the use of dairy cultures like the other cheeses worldwide. Halloumi cheese is unique. This is due to its versatile properties - a high melting point and a high pH, which means it can easily be fried or grilled. (KKTC Tarım Bakanlığı, 2015)

Cooking the curds after pressing is the first preservation method. Because halloumi cheese after pressing the curds cooking in the whey at 95 °C about 1 hour. Although this method kills all the pathogenic microorganisms in the curd and creates its specific structure.

Brine is the second preservation method which is salty whey added halloumi cheese before packing and the salt prevents the cheese until its expiry date.

The different chemical values in milk effect the diary products quality. The dry matter and the fat values in milk were low, the diary products' dry matter and fat content parameters decreased as well. Although the chemical values in milk has high parameters of fat and dry matter, the diary products' dry matter and fat content increased. Addition to this , when the fat contents and dry matter were increased in milk, halloumi cheese which was produced by that milk, their mouth feeling, odor and taste increased as well.

Different chemical values in milk means that different quality of products. Fat, dry matter and pH values cannot be same all the types of milk. The climate and geography, feeding, accomadations, lactation and species, age, milking frequency and health of cows effect the quality of raw milk so effect the quality of diary product.

The fat in milk is one of the quality parameter that we do all acceptance the raw milk. Milk caontains approximately 3.4% fat. Initially milk fat is secreted in the form of a fat globule surrounded by a membrane. Each fat globule is composed almost entirely of triacylglycerols and is surrounded by a membrane consisting of complex lipids such as phospholipids, along with proteins. These act as emulsifiers which keep the individual globules from coalescing and protect the contents of these globules from various enzymes in the fluid portion of the milk. Although 97–98% of lipids are triacylglycerols, small amounts of di- and monoacylglycerols, free cholesterol and cholesterol esters, free fatty acids, and phospholipids are also present. Unlike protein and carbohydrates, fat composition in milk varies widely in the composition due to genetic, lactational, and nutritional factor difference between different species.

Like composition, fat globules vary in size from less than 0.2 to about 15 micrometers in diameter between different species. Diameter may also vary between animals within a species and at different times within a milking of a single animal. The fat-soluble vitamins A, D, E, and K along with essential fatty acids such as linoleic and linolenic acid are found within the milk fat portion of the milk (MEGEP, 2007).

The quality of milk is important for producing halloumi cheese. For example one of them is pH value. pH value ranges from 6.4 to 6.8 and it changes over time. Milk from other bovines and non-bovine mammals varies in composition, but has a similar pH.

The quantities of the various main constituents of milk can vary considerably between cows of different breeds and between individual cows of the same breed. Therefore only limit values can be stated for the variations. Total components outside the water in milk are called dry matter. Other component distinctions; milk fat-free dry matter and fat in dry matter. Total components outside the water in milk are called dry matter.

Besides total solids, the term solids-non-fat (SNF) is used in discussing the composition of milk. SNF is the total solids content less the fat content. The mean SNF content is consequently 13.0 - 3.9 = 9.1 %. (MEGEP, 2012)

Seasonal transition periods, temperature, humidity and the effect of daylight are important. Depending on the seasons, the amount and variety of feed can change the milk yield and composition of animals. In general, the amount of fat is increased in winter, while the amount of milk decreases.

The increase in air temperature and relative humidity causes a decrease in short chain fatty acids (6-12 °C), especially in milk fat.

With the effect of seasons, it is seen that there are changes in the amount of mineral matter, protein and lactose except milk fat.

Lactation period is one of the most important effects that changes milk composition and amount. The first milk in this period is not a normal milk feature and it is defined as the colostrum called mouth milk. Colostrum continues for up to one week, starting from 3 days before delivery. In general, an increase in milk yield is observed in the first 5-6 weeks of the lactation period and a decrease is observed in the later period. In the first 1.5 months of the lactation period, a decrease was observed in the amount of protein and fat, while an increase was observed in the period until the last 8 - 8.5 months of the lactation period. Especially the level of fat is increasing more recently. While the amount of short-chain fatty acids decreases during the lactation period, the rate of unsaturated fatty acids is increasing. This also affects the melting point of milk fats. At the beginning of the lactation period the temperature of the curve is around 32 ° C. At the end of the lactation period, this temperature reaches about 53 ° C. There is an inverse ratio between the amount of milk and milk fat. As the amount of milk increases, the milk fat content decreases.

This situation shows itself better in lactation period. The amount of milk that increases during the lactation period decreases well towards the end of the lactation, while the amount of fat in this period is inversely proportional to this increase, while the amount of fat increases initially to the end.(Nateghi et al.,2014).

The aim of the study was to investigate the effect of seasonal changes on composition of

milk and halloumi cheese also on sensory attributes of halloumi cheese. Shanges in fat, dry mater, pH and sensory attributes (in halloumi cheese only) of these products were analysed during the production period of one year. Monthly weather temperature and humidity forecast data were also collected.

#### **CHAPTER 2**

#### THEORETICAL FRAME WORK

#### 2.1 Raw milk

The quality of incoming raw milk – and its bacterial content – affects more than just the processability of dairy products. It also has an effect on the quality of products when they reach stores, and their shelf life. Milk quality affects the efficiency and cost-effectiveness of plant's processing machinery, in particular heating units. Poor quality milk can clog heat exchangers, cause process interruptions and requires extra cleaning time – with all the costs associated with these two events (Shmidt, 2000).

A variety of tests are available to illuminate different aspects of milk quality and help the sort milk into different quality grades. Some of them may be more relevant to your dairy operations than others, depending on location, climate, equipment, supply chain and the others. There are some acceptance analysis in factories like alcohol test, temperature, fat, dry matter, pH value, antibiotics and aflatoxin M1. After theese analysis factory accepts the raw milk or rejects as shown in Figure 2.1.





Figure 2.1: Raw milk flow diagram

#### 2.2 Halloumi cheese

Halloumi cheese has two production types. Fresh and mature production.

#### 2.2.1 Fresh Halloumi

Rennet is added to milk and the product emerges with its unique production method and the properties of the following are given below.

### **Physical Properties:**

- Shape: Double-layer semicircle or double-layer rectangle
- Color: Light yellow to white
- Structure: Easily sliceable

### **Organic Properties:**

- Type: Semi-hard and elastic.

- Smell and Taste: From the feeding of dairy animals with plants grown in Cyprus taste and odor (less salty, slightly acidic)0

## **Chemical Properties:**

- Maximum humidity percentage: 46%
  - Minimum fat percentage: 43% of dry matter
  - Maximum salt percentage: 5%

## 2.2.2 Mature Halloumi:

Rennet is added to milk and the product emerges with its unique production method. After the halloumi cheese is put into salted whey to mature.

#### **Physical Properties:**

- Shape: Double-layer semicircle or double-layer rectangle
- Color: Light yellow to white
- Structure: Easily sliceable

#### **Organic Properties:**

- Type: Semi-hard, hard

- Smell and Taste: From the feeding of dairy animals with plants grown in Cyprus taste and odor.

#### **Chemical Properties:**

- Maximum humidity percentage: 37%

- Minimum fat percentage: 40% of dry matter
- Maximum salt percentage: 6%
- Maximum acid: 1.2% by dry matter as a milk acid (lactic acid)

Producing halloumi cheese, firstly pasteurize milk 72 °C 15 sec and after that cooling to 34–35°C. Second step is adding rennet or a similar enzyme according to the manufacturer's directions, while stirring the milk. After stirring the milk hold the milk at 40–42 °C until the curd sets. After coagulation, the curd is cut into 3–5 mm cubes using round knives in process. Hold the curd in whey for about 20 minutes, stirring gently and continuously, and then allow it to settle. Drain the whey and scoop out the curd into a hoop lined with cheese cloth. Press the curd. While the curd is in the press, heat the whey to about 80–90 °C. This precipitates the whey proteins, which can then be removed and pressed to make a whey cheese. Continue heating until the inside temperature become 85 °C every halloumi and its structures become soft and elastic. Remove the halloumi when still warm and rub in a little dry salt. When the pieces are cold, put them in vacuum packs filled with brine (Tarım Bakanlığı, Tescil, 2015).



Figure 2.2: Halloumi cheese production flow diagram

#### 2.3 Factors effecting the composition of milk

After milking changes that occur are usually microbiological. The chemical composition of milk can be varied by cooling, storage and applied technological processes. Also; drugs used in the treatment of diseases, antibiotics, pesticides, detergents and disinfectant residues, pregnancy and anger status, dry time and body secretions are also effective. Factors affecting the composition of raw milk before milking and during milking are:

a) Animal breed

The milk yield and the proportion of substances in the composition depend on the race. There may even be differences between different individuals of the same race. For this reason, breeding studies have been developed races giving high milk fat ratio.

b) Lactation:

The lactose ratio is generally constant during lactation. However, fat and protein fall in the first 3 months of lactation and increase towards the end.

c) Age of the animal:

The ability to synthesize milk is lost to a certain extent as the metabolism of the animal begins to lose weight. As a result, there is some decrease in the dry matter rate.

d) Health status of the animal:

The animal's health condition and diseases cause the animal to lose weight and decrease efficiency. In some patients such as mastitis, the composition of milk changes. In mastitis infection, microorganisms pass to alveoli. Milk-producing cells are damaged by their ability to synthesize fat, protein, and lactose.

Other changes in milk: Osmotic pressure should be kept at a certain level some ions in the blood go into the milk and the amount of chlorine in the milk increases. At the same time, the amount of lactose decreases. With the increase in the amount of chlorine increases the electrical permeability of milk. Partially damaged tissue passes into serum protein form and increases their amount. The amount of catalase enzyme and viscosity increase. The acidity

of milk decreases. Mastitis also creates problems in terms of technology. Microorganisms causing this disease include: *Streptococcus agalactiae, Streptococcus disagalactiae, Streptococcus salivarius, Staphylococcus aureus, Corynebacterium pyogenes, Clostridium perfringens, Aerobacter aerogenes.* 

#### e) Temperature (seasons)

Generally, optimum efficiency is obtained at 5 to 20 °C. Especially when the relative humidity is high with high temperature, decreases in oil ratio are seen.

#### f) Milking time and milking type

The milk yield of an animal increases with increasing the number of daily milking. Milking should be done 3 times a day on average. With proper milking, it acts as a massage to the mammary glands of the animal's breast and increases the yield. It is important to take the milk completely during milking.

#### g) Feed

During the synthesis of milk the necessary substances are taken from the body through the blood. If these substances are not given to the animal constantly, the animal meets the necessary substances from their own body and gradually weakens. After a while, the yield is reduced and the composition becomes poor. Animal's ability to benefit from feed is a genetic feature. Some of they convert food to better milk and meat. Green baits, corn and wheat bran, sunflower increase the yield of milk. Rape and sesame reduce yield.

#### h) Animal psychological status and care

In order to increase the efficiency in developed countries, a quiet barn environment is provided, and even the nerves of the soothing music are used. Inadequate poor care, noise, and insect discomfort reduce the yield. (Ozrenk et al., 2008)

#### 2.4 Determination of fat content

Fat is the most variable component (from 2.8 to 6.5%) of the milk, and has a higher commercial value. Its determination became almost a routine in the dairies, because the easiness of extraction of fat from milk.

## 2.4.1 Gerber method

#### **Apparatus and reagents**

- Gerber butyrometer for milk and appropriate corks;
- Shelf for butyrometer;
- Volumetric pipette (10 and 11 mL);
- Gerber centrifuge; Sulfuric acid (density 1.825 g/L)
- Amyl alcohol (density 0.815 g/L) Gerber's method

#### Procedure

- Carefully add 10 mL sulfuric acid in the butyrometer;

- Carefully add 11 mL milk to the butyrometer, by letting it to slowly flow down the glass walls in order to it does not mix with the acid;

- Add 1 mL amyl alcohol;
- Clean the neck of the butyrometer and close with cork;
- Agitate as inverting the butyrometer so the three liquids are mixed;
- Centrifuge for 4 to 5 minutes at 1200 rpm in the Gerber centrifuge;

- Remove the butyrometer of the centrifuge and adjust the meniscus to accomplish the reading.

The reading value in the scale is the result of the percent fat in the milk (% mass / volume)

#### 2.4.2 Van Gulik method

It is the fast and easy determination of the amount of fat by butyrometric method in cheese. The inclusion of non-fat components in the cheese with concentrated  $H_2SO_4$  and amyl alcohol, the ratio of fat (fat in 100 g of cheese) as determined by a special Van Gulik butyrometer.

Analytical scales (at least 0.1 mg in sensitivity), gerber centrifuge, water bath, Van Gulik cheese butyrometer and beaker, pipette and burette (1ml, 10ml), sulfuric acid (at 20 °C d<sub>20</sub>=  $1.522\pm0.005$  g/ml), amyl alcohol (at 20 °C d<sub>20</sub>= 0.808-0.818 g/ml)

Butyometer beaker is weighed 3 g at a sensitivity of 0.005 g from the cheese sample and placed firmly on the bottom of the butyrometer. 10 ml of  $H_2SO_4$  is placed from the top of the butyrometer and the top mouth is sealed with a special plug. Butyrometer 65-70 °C water bath in the occasional bottom of the cheese is expected to melt completely. After complete dissolution of the cheese, remove the top plug and shake gently, adding 1 ml amyl alcohol.  $H_2SO_4$  is then added to the line 35 at the neck of the butyrometer. The top of the butyrometer is dried with a small blotting paper and closed again with the plug. Shake well from bottom to up. Butyrometer is waiting in the water bath for five minutes. The centrifugal butyrometers are placed facing and centrifuged at 1000-1200 rpm for 10 minutes. The butyometers are kept in the water bath at 65-70 °C for 4-5 minutes. At the end of the procedure, the % fat ratio is read directly from the butyrometer scale. The result obtained gives % fat content of the cheese at 100 g.

#### 2.4.3 Soxhlet Extraction Method

Lipid in food present in various forms like monoglycerides, diglycerides, triglycerides and sterol and free fatty acid and phospholipid and carotenoids and fat-soluble vitamins. Lipid is soluble in organic solvent and insoluble in water, because of this, organic solvents like hexane, petroleum ether have the ability to solubilize fat and fat is extracted from food in combination with the solvent. Later the fat is collected by evaporating the solvent. Almost all the solvent is distilled off and can be reused.

#### **Preparing the sample**

First of all, we have to dry the product and remove moisture in order to facilitate entry of the organic solvent, because moisture restricts the entry of organic solvent. Then size reduction is there to increase the surface area and due to it, there is larger exposed surface. After this, we go for acidic hydrolysis which helps in breaking of protein fat emulsion and increases the availability of fat for the solvent. Furthermore, we can collect the solvent by distillation.

#### **Apparatus and reagents**

- Weighing balance
- Soxhlet apparatus
- Drying oven
- Thimble
- Heating mantle
- Glass rod
- Desiccator with silica gel
- Petroleum ether (Boiling temperature 60°-80°c)
- Cotton plugs



Figure 2.4: Apparatus Used For Soxhlet Extraction Method

#### **Procedure:**

- First of all, rinse all the glass apparatus by petroleum ether and dry it in the oven at 102°c and after removing it keep in the desiccator.
- 2. Weigh 5 gram of grounded and dried sample and place it in the thimble.
- 3. Place the thimble in the soxhlet extractor.
- 4. Take a 150ml round bottom flask and clean it and fill the flask with 90 ml petroleum ether.
- 5. Place the whole setting on a heating mantle and allow the petroleum ether to boil.

- 6. Continue the extraction process for several hours, almost 6 hours.
- Remove the condensing unit from extraction unit and allow the sample to cool down. Finally, it removes all the lipid.
- 8. Collect almost all the solvent after distillation.
- 9. Place the sample in the oven and after removing it place in the desiccator.
- 10. Take the weight of the sample.
- 11. As a result, we get a defat sample.

#### **Calculation:**

Empty thimble=  $w_1$ 

Thimble with sample=  $w_2$ 

Weight of sample= p

Then crude fat percentage ig=  $(w_2-w_1)/p \times 100$ 

This method is an efficient method to extract all the fat present in the food. Hence it is used in oil extraction units for better recovery of oil. This method is also applied to the deoiled cake which is collected from screw impellers rather than high-pressure expression. It is also used in the analysis of fat present in the sample.

#### 2.4.4 Photometric methods

In order to determine the amount of fat in milk by photometric method, tools similar to those described in the milkotester automatic fat analyzer were developed.

The milkotester automatic device has been widely implemented for routine analysis in recent years due to the large number of samples analyzed in a short time.

### Application area

Whole milk, homogenized milk, skimmed milk, whey, cream. principle of this machine: Place the milk sample beakers on the bant. These beakers move forward automatically every 30 seconds. A mixer mixes the samples thoroughly, then 2 ml of milk is sucked through the milk pipette and the sample is transferred to a milk reservoir. Wherein a sprayer is mixed with a metered amount of dilution solution, which is solvent-soluble in the milk proteins. Then the mixture is homogenized in the high-pressure pump so that the desired size of the fat beads is reached. The homogenized mixture is sent to a micro cuvette. The optical density is measured by means of a photocell. As the light beams transmitted pass through the sample, this transition is more or less spread depending on the number of fat beads. The resulting turbidity is measured photometrically. The percentage of fat in the display is expressed directly up to 1/100 in numerical order. Results are passed on the strips or cards in a writing machine connected to the instrument.

The error limits on milkotester automatically increase as the fat level of the sample increases. At %2.2 fat grade, it varies from  $\pm 0.005\%$  to  $\pm 0.011\%$ , at %8 fat grade  $\pm 0.2016\%$  to  $\pm 0.041\%$ . According to these data; the milkotester automatic device is found to be more sensitive than the Gerber method, which has a margin of error of 0.05%

#### 2.4.5 Infrared – spectrophotometric methods

**Milkoscan:** This electronic instrument provides us the ability to quickly analyze milk samples for components (i.e. fat, protein, lactose, total solids and solids non-fat.). In basic terms, the instrument provides milk component results through infrared light measurement.

#### 2.5 Determination of dry matter content of milk

- Determination of water soluble dry matter,
- Determination of dry matter in water,
- Determination of total dry matter (drying method in the oven).

#### 2.5.1 Water Soluble Dry Matter Determination

Water-soluble dry matter is an important criterion in both production and quality control. The amount of water-soluble dry matter;

In determining the maturity and harvest time in fruits,

Fruit juice, concentrate, tomato paste or canned food processing is constantly important in keeping control of the stages.

Determination of water soluble dry matter; The refractometer can be made by using various

methods with the help of the isometer (brix hydrometer) or drying cabinet.

Working principle of refractometer; The optical density is based on the refraction law and the breaking of light as it passes from one medium to another in different environments.

Fat-free dry matter can be read directly with refractometers. When making an evaluation according to the total amount of dry matter, the following criteria should be considered:

a-) Species and race: is the factor that has the most effect on dry matter. Studies have shown that the dry matter in the milk of different species and racemates is different.

b-) Yield: When the yield is high, the amount of dry matter decreases, and towards the end of lactation.

c-) Age: the amount of dry matter decreases slightly in the milk of advanced animals.

d-) Season: Although there is a decrease in the dry matter in the summer months, there is an increase in winter months.

e) Sickness: Mastitis and other breast inflammation cause the amount of dry matter to be reduced.

f-) Nutrition: Despite the decrease in milk yield in animals not fed enough amount of dry matter is seen.

g-) The effect of the substances added to milk or milked: The substances added to the milk for the purpose of cheating, such as water, skimmed milk, submarine, whey and the like, reduce the amount of dry matter, milk powder, cream, darkened milk, soda etc. substances increase the amount of dry matter. It does not affect the fat-free drying of milk, but also reduces the dry matter.

The dry matter scale in the Abbe refractometer was prepared according to the pure sucrose solution at 20 ° C. Besides the refractive index scale, there is also a dry matter scale.

Thus, after each reading, there is no need to apply to the tables with% water soluble dry matter content. In addition, it is a kind of refractometer which can be used to circulate water in prism liners, so that reading can be done at a fixed temperature.

#### Measurement with refractometer

The necessary lighting is provided by a suitable and sufficient light source.

The prism cap is opened and checked for clean. If necessary the prism is washed with pure water and dried by wiping with a soft tissue.

2 - 3 drops are applied to the clean and dry prism surface at the room temperature. The sample is dropped from above without touching the surface of the prism.

After the sample is installed, the prism cap is closed.

With the adjustment screws, the light and dark areas are clarified.

Read the figure at the intersection of the dark and bright area.

After the reading is done, the prism surface is cleaned and dried directly with pure water.

Soft paper is placed between the prism and the lid without removing the special packaging.

Before using the refractometer, check whether it is set. If 0 "is read when pure water is added to the prism, the refractometer is set.

#### Calculation

In any non-reconstituted example, the value read in the refractometer gives directly the percentage soluble dry matter ratio. There is no need to make any calculations. As in the case of marmalade, dilution should not be applied unless it is mandatory. When reconstitution, calculation is made using the following formula.

Water Soluble Dry Matter %(g/100 ml)=  $\frac{BxV}{S}$ 

B= Brix V=diluted volume (ml) S= sample (g)

#### **2.5.2 Total Dry Matter Determination (Drying Method)**

For the determination of the total dry matter, generally the drying method is used in the oven. Furthermore, the distillation method and the electrical conductance method can be used to determine the dry matter. Although the electrical conductivity method is very fast, it is preferable to use the drying method in the oven.

#### **Process steps**

Dry matter containers are dried at 105°C and brought to constant weight.

Dehydrated in desiccator is cooled.

5 to 10 g or ml of the sample is weighed or pipetted from the liquid or solid food material into the dry matter containers.

If solid food sample directly to the desired degree the meat is set (105 °C for meat, 130-135 °C for flour).

If the sample is liquid, it is kept on the water bath until the water has flown and then the meat is dried and dried to the constant weight (same result in weighing).

When working with samples such as cheese, the tare of dry matter containers is taken with some sand and a small baguette. The sand facilitates both the crushing operation and the expansion of the surface, facilitating the outlet of water in the environment.

The dry matter containers removed from the oven are weighed after cooling in the desiccator. The process must be repeated in at least two parallels and the difference between the parallels should not exceed 0.1.

m<sub>1</sub>: Dried empty drying container and weight of the lid (g)

m<sub>2</sub>: The weight of the drying container and its lid before drying (g)

m<sub>3</sub>: Inside the sample, the weight of the drying container and lid after drying (g)

% dry matter=  $\underline{m_2 - m_3} \times 100$  $\underline{m_2 - m_1}$ 

#### 2.5.3 Cryoscopy

The cryoscopy measures the freezing point of the milk and it is the most effective method for the determination of faking by addition of water. The freezing point is directly related to the concentration of its water-soluble constituents (lactose and mineral salts). To determine the cryoscopy, the electronic cryoscope is used for analysis of the milk, due to its reliability, although the high cost of process is well known. For this reason, this analysis is only used for average scale.

#### **Apparatus and reagents**

- electronic cryoscope
- specific tubes
- freezing solution 0.422
- freezing solution 0.621

#### Procedure

Calibrate the device with the freezing solutions before beginning the analyses.
Take out
2.5 mL milk of the sample to be analyzed and place it into the appropriate cryoscope tube.

- Clean the electrode of the equipment with absorbent paper
- Switch on the equipment
- Wait for the thermal sign of the analysis

- Read on the digital display. The result indicates either the value of the freezing point depression in Hortvet degrees and the percent water. (Bradley, 2010)

#### 2.6 Methods of determine acidity

The milk shows acidic reaction, which is called first acidity or natural acidity. This acidity consists of first order composition of casein, phosphate and citrates; Second degree comes from albumin, globulin and carbon dioxide. Milk cannot maintain its first acidity for a long time. Due to milking and holding conditions, different kinds of microorganisms are transmitted to the milk in various ways. The activity of these microorganisms causes acidity in milk. Some acid-producing bacteria, especially lactic acid bacteria, break down milk sugar into lactic acid and cause acidity to increase. As such acidity develops later, it is called acidity or developing acidity. Total acidity in milk consists of the first acidity and acidity.

Total acidity = Natural acidity + Improved acidity

The degree of acidity of the milk is carried out to determine whether it is fresh and normal, whether it can withstand the temperature during processing, whether a neutralizing agent is

added, whether the milk is mastitis, whether it is in conformity with the bylaws, standards and codes. Durability tests such as titration acidity, alcohol test and boil test are applied to determine the increase in acidity in milk. A relationship is established between the level of acidity and hygiene, and the degree of acidity helps to decide which product is suitable for milk. Acidity is considered as a quality measure in some dairy products. It also helps to direct the production of some dairy products.

There are several methods for the determination of acidity in milk. These;

- Boiling test
- Alcohol test
- Litmus Examination
- Titration
- pH

### 2.6.1 Boiling Test

It is a simple method to determine if raw milk is fresh or milk is suitable for heat treatment in places where no analysis is possible.

Fresh and normal milk does not coagulate when boiled. However, acidity increased milk; with the effect of the temperature, the casein micelles neutralize more quickly and the colloidal state of the micelles deteriorates and clotting occurs. The development of acidity can be determined roughly.

#### Apparatus

Fire resistant glass tube, erlen

#### Procedure

After some milk is placed in the container, milk is boiled on the bunzen. The structure of milk is visually inspected and checked for clot formation.

#### **Evaluation of the result**

At the end of the boiling process it is understood whether the milk is clotted by heat treatment.

## 2.6.2 Alcohol test

It is done to determine whether the acidity of milk increases.

### Usage Area

Due to its rapid results, more milk is mixed with 68% alcohol during the acceptance of the product and it is among the daha platform tests.

For patients and children, to determine the acidity of the less developed milk; 2 parts 68% alcohol is mixed with 1 part of milk and milk with less than 8 °SH degree is distinguished. This is called. Double alcohol test 'or' double alcohol test.

If the milk is brought from a very remote location; In order to increase the sensitivity in the alcohol test performed in milk collection centers, alcohol with high concentration should be used as 70%, 72% or 74%. This reduces the danger of deterioration of acidity during transportation.

## **Preparation of the sample**

The sample should be mixed thoroughly. During the measurement, the milk sample temperature should be approximately 25 °C. Performing the test In a test tube, 5 ml of milk and 5 ml of alcohol are placed and shaken strongly.

- As a result of the alcohol test, no clot in the milk is considered as proof that the acidity is less than 8 °SH or the pH value is higher than 6.4 and the milk passes the alcohol test. In other words, milk is suitable for heat treatment.
- Formation of uncertain and very small clots, acidity of 8-8.5 °SH;
- The formation of small clots indicates that the acidity is between 9.0-10 °SH.

• If a clot occurs in the milk during the platform test, the milk is not accepted and returned to the manufacturer.

• Colostrum is highly clotted as a result of alcohol testing, even if the acidity of milk with severe mastitis is normal.

#### 2.6.3 Litmus Examination

Turnosol indicator gives red in acid and blue in alkaline. The reaction in fresh milk is amphoteric. If the blue litmus is very red, it is understood that the acidity is high.

#### 2.6.4 pH Measurements

The most accurate result of the dissociated part of the acidity is the pH value. Information about the amount and activity of the free hydrogen ions in the medium is obtained by the pH measurement and this acidity is called the actual acidity. The pH value gives important clues about the yield and value of the product.

#### The principle of the method

Device called pH-meter; It is a device with two electrodes connected to the electrode (voltmeter) which contains a combined single electrode or glass-cube double electrode, the pH is measured by dipping into the solution to be determined. Electrodes; It has the property of detecting the positive charges in the solution and resulting from hydrogen ions. These charges are collected on the electrodes and transmitted to the very sensitive potentiometer and the result is read as the pH value from the scale.

#### Apparatus

pH-meter: Measuring range 0-14 pH

#### Procedure

First, the pH-meter is adjusted with the help of buffer solutions. Then the electrode system of the pH meter is plunged into the milk sample. After the process is finished, the electrode is carefully cleaned with distilled water at about 50 ° C and dried with filter paper. If necessary, wash thoroughly with special cleaning solution and dry.( Coşkun and Çağlar,1997)

#### 2.7 Methods of sensory analyses

### 2.7.1 Single Sample Evaluation

Used to determine consumer preferences. Foods find the market when they are produced in accordance with consumer requirements and specifications.

Therefore;

- New product development
- Formulation development
- Increase diversity in the product

Sensory evaluations are made to consumers in order to increase market share.

Only the sensory characteristics of food can be controlled by consumers. Consumers' reactions to the appearance, taste and texture of food are determined in the determination of consumer preferences.

#### **Application of Single Sample Evaluation**

- 1. An example is presented to the consumer.
- 2. About the product given from the consumer;
  - Accepting or rejecting the Product
- Describes the rating of the product
  - Points to the product
  - Define the perceptions of the product with his own sentences
  - The product is asked to define it in the recommended terms.
- 3. Results from consumers are evaluated by analysis of variance.

#### **2.7.2 Differential Tests**

The reasons for the differences in sensory properties of foods are;

- Genetic properties

- Soil, cultivation and climatic conditions of crop products

-Feeding and maintenance conditions of animal products

-Applications before and after slaughtering of animal products

- Food processing method

-Type of material used in packaging and packaging

-Changes in storage and marketing conditions

Differential tests; It is applied in order to determine the sensory differences that occur as a result of physical and chemical changes in foods during production, storage and marketing stages and is based on the perception of difference between two or more samples.

#### Application of the difference tests:

- Preparation of quality standards

- Development of quality control programs

-Preparation and elimination of samples for consumer preference testing

Panels applied by the difference test technique are asked to the following questions:

-Is there a difference between samples?

-What is the density and / or direction of any difference between samples?

The difference tests are classified as follows:

#### Single Stimulus Test (Binary test)

It is also known as A-A test and is mostly used to determine consumer preferences. Two examples are given to the taster. One of these examples is the standard sample (control sample, reference sample), the other is the sample (test sample) to be evaluated.

Standard example: Examples of food or drink consumed by the consumer, known and loved.

Experiment example: Examples of food or beverage similar to the standard sample in terms of the quantity and variety of the ingredients in the composition or the production process.

#### **Duo-Trio Test**

#### **Application of the test**

1. The panelist is asked to evaluate the standard sample first.

2. Then two different test samples are presented. These two samples are asked whether they are similar or different to the standard.

The double-triple test is applied to improve the quality and control of newly produced products.

#### **Triangle Test**

It is one of the most widely used difference tests. The triangle test differentiation tests are also used for panelist selection.

#### **Application of the test**

1. There are three examples at the same time. At the same time, two different examples of the same one are given for panel evaluation (standard sample is not used).

2. It is desirable to identify those that are similar or different from the given examples.

## **Matched Benchmark Test**

#### **Application of Matched Benchmarking Test**

1. The panelist is asked to evaluate both the standard sample and the test sample at the same time.

2. About the product given to the panelist; the two samples are asked whether they are the same or which are better in terms of sensory properties such as flavor, appearance, odor, whichever is more watery, which is more sweet.

#### 2.7.3 Quality-Quantity Tests

Quality-quantity tests are the tests where one or several sensory quality characteristics of foods are evaluated by different techniques depending on the variety and density. By applying quality-test;

Determine whether the quality of difference in the examples.

The degree of difference is determined.

Choosing the best example or samples is provided.

The samples are determined whether there is any improvement or deterioration.

Samples are sorted in terms of quality, color, flavor, etc., and evaluated in terms of total quality or acceptability.

More than three samples are presented to the panelist in the quality-test. In these tests, 20 samples can be presented simultaneously in one session. However, a maximum of six samples should be presented to prevent the sensory organs of the panelists from getting tired and reducing their attention.

#### **Sequencing Test**

1.Panel is given many examples at the same time.

2. It is desirable to rank the given samples according to the order of preference or the intensity of the sensory feature.

3-Results of the panelists are evaluated by analysis of variance

#### **Scoring Tests**

It is a technique used for grading the quality characteristics of a sensory properties of samples such as color, texture, flavor by using a numerical or special scale or measuring the density of these quality characteristics. The scores given by the panelists in the scoring tests are averaged.

Scoring tests can be carried out with at least three trained panelists. However, it is recommended to apply with a large number of panelists. For the scoring test to be effective and reliable, the following should be done:

A realistic scoring should be made that can clearly and fully demonstrate the quality characteristics of the food.

Quality characteristics to be scored on the test card;

Appearance features

Textural features

Odor properties

Taste properties should be listed.

Understanding and understanding of the meaning of each score among the panelists consensus in terms of evaluation.

Among the points to be used,

There should be enough difference to indicate the variation.

#### **Application of Scoring Test**

1. Panel is given as a single or many examples.

2. The given sample (s) are asked to score according to the order of preference or the intensity of the sensory feature.

3-Results of the panelists are evaluated by analysis of variance

#### **Hedonic Scale Method**

Hedonic scales are mostly applied to consumer tests and uneducated panelists.

Hedonic scales can be used to evaluate the preference or dislike / dislike of panelists. Hedonic scales can be prepared as verbal, facial expression, graphic (linear).

#### **Profile Tests**

Generally, it is the technique applied with good panelists trained in sensory analysis of foods with wide sensory properties. In profile tests, 3 puan8 panelists discuss common or individual points around a table, for example by discussing their sensory characteristics.

Then the points given are transferred to a chart. It can be texture or flavor profile.

#### a. Texture Profile

Mechanical, geometric textural properties of foods, fat and moisture properties and density of these properties, the intake of foods from the mouth until the swallowing of the changes in the stages of the sensory analysis method is evaluated. Texture Profile Analysis 5 .7 trained panelists and panelists will evaluate the textural properties of foods with standard grading scales.

#### **b. Flavor Profile**

It is the technique in which all food-related properties are evaluated in detail with the best trained panellists. In flavor tests, the flavor characteristics of foods are evaluated. However, in flavor profile analysis, the relationship between the compounds that make up the flavor and all the flavor of the food is evaluated.

- Taste profile analysis is carried out for the following purposes:
- Increase sales, increase market share
- -Develop or re-formulate
- -Developing new products to meet consumer specifications

#### **Robinson Sensory (Organoleptic) Test**

It is one of the tests to check the suitability of food packaging. The effect of the printing material, ink or lacquer used in the packaging on the sensory quality of the food can be determined. This test method involves determining whether there is a sensible difference in taste and odor between the two product samples. Robinson sensory (organoleptic) test is performed by using 10 experts and 10 materials. As reference sample; If the printing material is to be tested, the product to be packaged, ink or lacquered is considered to be wrapped with unprinted material. The taste and odor characteristics of the samples to be tested together with the reference sample are evaluated by the subjects. (MEGEP, 2012; Watts et al., 1989)

#### **CHAPTER 3**

#### **RELATED RESEARCH**

Papademas and Robinson (1998) have studied Halloumi cheese and its characteristics in 1998. They reported that halloumi, Traditional cheese of Cyprus is quite popular in the Middle East and the Mediterranean regions of Europe. The process of making the basic cheese puts Halloumi into the cheese family, but some of the features are quite unique. In his articles, studies on chemical composition, production procedures, sensory quality and Hellim storage were reviewed and some proposed improvements, such as the use of diluted milk powder or homogenized milk, were discussed.

Economides et al. (1987) have studied the effect of different milks on the yield and chemical composition of halloumi cheese. They said that the halloumi cheese was made from milk (sheep, goat and cow) and equal parts of sheep and goat milk mixture. The chemical composition of all milk, cheese and cheese by-products was determined. Sheep milk was the highest, cow milk had the lowest fat, protein and total solids content. The same substances were also determined in the first and second whey. When pure sheep milk was used, the production of curd and / or halloumi cheese was highest. The milk required to produce a kg of Halloumi cheese was 5.44,8.85,11.30 and 6.70 kg for sheep, goat, cow and mixed (sheep and goat) milk, respectively. Fat and protein recoveries were sufficient for sheep's milk, but were considerably lower for all other milk. Multiple linear regressions were used to develop predictive equations for cheese production from all four types of milk. Fat, protein, casein and casein / fat ratio were important variables in predicting the output of cheese. The accuracy of the estimation was still high when only two variables were used in the regression equations which were the same in all the milk (fat and protein) except goat milk (fat and casein).

Atasever et al. (1999) have studied halloumi cheese samples produced from 1: 1 sheep and cow milk mixture. Cheese samples were divided into two groups. Samples of the first group

were packaged in dry saline and then in vacuum. Subsequent group samples were matured in lin packages containing saline solution containing 13% salt. In order to detect changes in chemical, microbiological and sensory properties, samples were examined during maturation period. The dry matter and fat content and acidity values of the first group of samples were higher, but the salt and ash contents were lower than the samples in the second group. In addition, salt and ash content and acidity values increased and pH values decreased in both groups during ripening period. From a microbiological point of view, coliforms, total viability, yeast and mold count were higher in the first group than in the second group. During the maturation period, coliforms were decreased in both groups. On the other hand, the number of tolal organisms, yeast and molds decreased in the second group. According to sensory evaluations, first group samples were more preferred. Cheese yield was determined as 15.40%.

Kamleh et al. (2012) have studied Halloumi cheese blocks packed in vacuum polyamide / polyethylene laminate sachets stored at 5, 15 and 25 ° C. Total bacterial count, lactic acid bacteria, total anaerobic bacteria, yeasts and molds, pH and titratable acidity changes were observed during storage. The appearance of the packed Halloumi cheese significantly correlated with the number of different microbial populations living in cheese. The shelf life of the stored Halloumi cheese was evaluated using the survival analysis and the consumer's rejection index. The nominal shelf life of Halloumi cheese was 79.6, 37.8 and 2.6 days when stored at 5, 15 and 25 °C, respectively. The  $Q_{10}$  values (shelf life at T °C / shelf life at T + 10 °C) were 2.1 and 14.5 at 5 °C and 15 °C, respectively. The increase in the number of different microbial populations during storage stresses the need for good production practices and low temperatures during the storage and distribution of packaged Halloumi cheese.

According to halloumi registration (2015), milk used in the production of halloumi; all or most of the feed needs of dairy animals are met by the rough feeds produced from free grasslands and indigenous plants in Cyprus and it has unique taste and odor. It is not allowed to add milk powder or thickened milk, rock salt, colorant, preservative or other additives into the skin. Also in halloumi production the presence of antibiotics, plant medicines and other harmful chemicals in the milk is prohibited. Sheep and goat milk was provided by native

breed animals and hybrid animals adapted to the climatological conditions in Cyprus. To be more precise, sheep milk was obtained from the flocks of Cyprus native sheep with thicken tails and the breeding of these sheep with the breeding programs of the Sakız, İvesi, and Ost-friesian races. Goat milk is obtained from native breed goats and their hybrids with Damascus goats spread over the whole island. Cow's milk is derived from the black-hull (Holstein Freisian breed) cows, which were gradually introduced to Cyprus starting from the last hundred years, and were very well adapted to the climatic conditions of Cyprus. Cows are made from concentrate feeds produced in Cyprus, most of them with mixed feeds in pellet and powder form, and roughage (green, dry grass, silage and straw form) which are grown in Cyprus fed. Halloumi cheese shape was double-layer semicircle or double-layer rectangle, its colour was light yellow to white color and easily slicable. Its structure was semi-hard and elastic, specific taste and odor (less salty, slightly acidic) mouth feeling was caused by the feeding of dairy plants in Cyprus. Its moisture was 46%, fat amount was 43% of dry matter and 5 % of salt.

#### **CHAPTER 4**

## MATERIALS AND METHODS

#### 4.1 Materials

Milk and halloumi samples have different fat value, ph value and dry matter. All samples analyzed one by one. Also in one year (four seasons) halloumi cheeses analyzed by quality-quantity tests per month.

#### 4.1.1 Preparing of milk samples

Raw milk samples were taken from the milk tank for analysis of ph value, fat value and dry matter. Samples taken for four times from each milk tank.

#### 4.1.2 Preparing of halloumi samples

Halloumi cheeses produced from the milk samples selected on the same day are shredded for analysis. The shredded cheeses are respectively analyzed for ph, fat and dry matter analysis and are separated from the same production sample to perform the analysis again 18 °C.

Halloumi cheeses prepared for sensory testing are only sliced.

#### 4.2 Reagents and other materials /equipments

**Gerber centrifuge:** The centrifuge that has heating to 65 °C, 8 compartments and time setting. The cycle was 1000-1200 rpm

**Butyrometers:** Two types of butyrometers were used for analysis. Cheee butyrometers and milk butyrometers for fat analysis. The butyrometers are glass and they must have rubber plugs.

**Pipets:** they are used for adding milk, amyl alcohol and sulfuric acid as well. Three types of glass pipets used in analyses. One of is 11.75 ml milk pipet for adding milk. one of is 10 ml for adding sulfuric acid and one of is 1 ml for amyl alcohol.

#### Sulphuric acid

The acid to be used in the gerber method should be in 90-91% of purity. 90-91%  $H_2SO_4$  with a density of  $1.818 \pm 0.003$  g/mol was used for milk, however 90-91%  $H_2SO_4$  with a density of 1.5 g/mol were used for cheese fat analysis.

#### **Amyl alcohol**

The amyl alcohol to be used for the analysis should be pure and its density should be 0.811  $\pm$  0.003 g/mol at 132° C

#### 4.3 Methods

#### **4.3.1** Determination of fat content of milk

Milk butyrometers are placed on supports with their mouths pointing upwards. Then 10 ml of sulfuric acid adding first and 11 ml of sample adjusted to  $20^{\circ}$  C is added with pipet on the inner wall of bottom tube of the butyrometers. Finally we add 1 ml of amyl alcohol. The butyrometer's lid is thoroughly and tightly sealed with a dry rubber plug. If the adjusting bracelet is used, it must be ensured that the metal bracelet is firmly placed in the throat of the butyrometer and firmly tied.

The butyrometer is slowly shaken and overturned until the existing clot is completely dissolved and no whiteness remains in the butyrometer. The butyrometer heats up during the dissolution of the clot. For this reason, a clean rag or shake bridged stand, or a rinsing machine can be used to be protected against temperature and possibly the risk of fracture of the butyrometer. During the analysis, goggles or even proper clothing should be used.

The butyrometers are then placed on the heating appliance centrifuge table in such a way that the plugs face outwards. To ensure equilibrium with centrifugal ratios, equilibrium must be established by placing a pre-calibrated butyrometer, which can be used in the same way as the counter-sample butyrometer or sample weight.

The batch of the butyrometers is centrifuged for 5 minutes at a centrifugation rate of 1000-1200 rpm with the graduated portion coming down.

In order to perform read at the bottom the stopper must be pushed by turning the stopper in the mouth to bring the lower limit of the fat column to one of the degree lines so that reading can be

done. Each section of the butyrometer corresponds to 1 g of fat in 100 g or 100 ml of milk, depending on the amount of sample taken. When 11 ml of the sample was taken, the result will be g fat/100 g milk.

#### 4.3.2 Determination of Dry Matter of Milk

The necessary lighting is provided by a suitable and sufficient light source.

The prism cap is opened and checked for clean. If necessary the prism is washed with pure water and dried by wiping with a soft tissue. 2 - 3 drops are applied to the clean and dry prism surface at the room temperature. The sample is dropped from above without touching the surface of the prism. After the sample is installed, the prism cap is closed. With the adjustment screws, the light and dark areas are clarified. Read the figure at the intersection of the dark and bright area. After the reading is done, the prism surface is cleaned and dried directly with pure water.

#### 4.3.3. Determination of pH value of milk and halloumi cheese

Liquid samples such as milk can only be tested by immersing the electrode into the sample without further processing.

Since the pH of cheese samples will vary from one piece to another, these samples are thoroughly grinded or crushed to make them homogenous. Take a sample from this homogenized halloumi cheese and place it in a small container where the electrode can easily contact. Electrodes are placed in the cheese sample with a thermometer for gentle placement of the electrodes. Cheese should not be diluted with water. This process will raise the pH up to 0.3 units because it will disrupt salt balance. Before testing the halloumi cheese, the pH meter is switched on and left running for 5 minutes. Then its accuracy is checked against standard buffer solutions. The pH range of standard buffer solutions for fermented dairy products and cheese should be between 4-6. Then the electrode is placed in the cheese sample. The pH value is read from the pH meter. After each determination, the sample container is moved and the same cheese sample is measured in three different places. This means that the correct measurement has been made. Then, after the electrodes are thoroughly washed and dried with blotting paper, another measurement can be made. If not, replace the electrodes.

#### 4.3.4 Determination of fat content of halloumi cheese

3g cheese is placed in the butyrometer by weighing the beaker placed in the bottom plug of the butyrometer. 10 ml of the sulfuric acid is placed in the open mouth. It is immersed in a water bath of 70  $^{\circ}$  C, shaken occasionally, until the cheese is completely dissolved. Then 1 ml of amyl alcohol is added and shaken and made up of sulfuric acid of the same specific gravity up to 35 parts of the butyrometer. The butyrometer is centrifuged for 10 minutes by closing the mouth with a rubber stopper. It is kept in a 65  $^{\circ}$  C water bath for 5 minutes and the amount of fat is read as % of the butyrometer scale.

#### 4.3.5 Determination of dry matter of halloumi cheese

3 g of grinded halloumi cheese sample is weighed, with the help of glass baguette thoroughly mixed with sand is spread to the bottom of the container.

Containers containing the halloumi sample are placed in PRECISA XM 60 at 105 °C and dried for a minimum of 30 minutes. Then the numbers on the screen indicate the % dry matter.

#### 4.3.6 Sensory evaluation of halloumi cheese

Quality-quantity tests are the tests where one or several sensory quality characteristics of foods are evaluated by different techniques depending on the variety and density. By applying quality-test;

The degree of difference is determined.

Choosing the best example or samples is provided. The samples are determined whether there is any improvement or deterioration.

Samples are sorted in terms of general appearance, color, mouth feeling, odor and taste and evaluated in terms of total quality or acceptability of halloumi cheese 12 different productions were measured by using a 5-point hedonic scale. In total, 10 panelists participated in the study.

Twelve different halloumi which were different months production cheeses, namely the months of the year January, February, March, April, May, June, July, August, September, October, November, December made by well-known manufacturers were used as samples.

They were packed in pet coex. The respondents tasted all samples coded with letters. Their sensory quality was assessed by an in-house panel.

A total of 10 consumers were recruited for participating in the study. They originated from different countries and were aged between 25 and 58 years, with a total of 5 females and 5 males.

The procedure was explained to the panelists. Halloumi cheese samples were served on white plates. They were advised to rinse their mouth with water before evaluating the consecutive sample. The panelists were asked to use a 5-point hedonic scale (1= not satisfactory; 2=fair, 3=medium, 4=good, 5=excellent) to evaluate the appearance, colour, flavour and mouthfeel of the twelve halloumi cheese varieties. They were also asked to select their preference for the general quality attributes of the halloumi cheese samples on a 5-point linguistic scale, (i.e. 'not at all important', 'important', 'highly important' and 'extremely important').

It should be possible to calculate one number that is typical of the whole data sample- a measure of location- and another number which indicates the spread or dispersion of the measurements. The simplest measure of location is the arithmetic mean, often simply called the mean of the sample (x). By far the most widely used measure of dispersion is the standard deviation of the sample (s). The standard deviation is used in many calculations. It may be calculated from the results of different measurements applying somewhat different equations. It is often useful to give the standard deviation of a sample as a proportion of the mean. This is usually expressed as a percentage, i.e. and is called the relative standard deviation (RSD) or the coefficient of variation (CV).

In this study, we calculated the individual means of each sensory attribute in order to be able to rank each halloumi cheese and also the general mean of each different months halloumi variety for overall acceptability.

#### **CHAPTER 5**

#### **RESULTS AND DISCUSSION**

For this studies, milk and halloumi cheese sample were taken during theone year production season. Samples were analysed in laboratory. Fat analyses, dry matter analyses and pH analyses made both of them. In addition to this, in a year (12 months) every months samples were taken and made sensory analyses for seeing the seasonal changes in halloumi cheese.

#### 5.1 Fat Analysis Results of Milk

The results belongs to monthly milk samples throughout for 12 months with 3 replications sample were summarized at Table 5.1.

When we look at the fat values of milk samples, they were changing in a range of 3,22-3,78 %.

Sample (months)	Fa	at amount %		Ave	SD	
January	3,8	3,8	3,8	3,78	0,03	
February	3,7	3,8	3,7	3,72	0,03	
March	3,6	3,6	3,5	3,55	0,05	
April	3,4	3,5	3,4	3,42	0,03	
May	3,4	3,3	3,3	3,32	0,03	
June	3,2	3,3	3,2	3,22	0,03	
July	3,3	3,3	3,4	3,32	0,03	
August	3,4	3,4	3,4	3,38	0,03	
September	3,5	3,4	3,5	3,47	0,06	
October	3,6	3,6	3,6	3,56	0,01	
November	3,7	3,6	3,7	3,63	0,03	
December	3,8	3,8	3,8	3,78	0,03	

Table 5.1: Fat amounts of milk samples during the processing period



Figure 5.1: Fat amounts of milk samples during the processing period

# 5.2 Dry Matter Analysis Results of Milk

The results obtained fresh milk dry matter values for 12 months in 3 replications at one time were summarized at Table 5.2.

When we look at the dry matter values of milk were found different every month. Although three replications were closed to each other every month.

Sample (months)		Dry matter value		Ave	SD	
January	9,8	9,8	9,8	9,8	0	
February	9,8	9,8	9,8	9,8	0	
March	9,7	9,7	9,7	9,7	0	
April	9,6	9,6	9,6	9,6	0	
May	9,6	9,6	9,6	9,6	0	
June	9,5	9,5	9,5	9,5	0	
July	9,6	9,6	9,6	9,6	0	
August	9,6	9,6	9,6	9,6	0	
September	9,7	9,7	9,7	9,7	0	
October	9,7	9,7	9,7	9,7	0	
November	9,7	9,7	9,7	9,7	0	
December	9,8	9,8	9,8	9,8	0	

Table 5.2: Dry matter of milk samples with standard deviation and average



Figure 5.2: Dry matter of milk samples with standard deviation and average

# 5.3 pH Value Analysis Results of Milk and Halloumi Cheese

The results obtained fresh milk and halloumi cheese pH values for 12 months in 3 replications at one time were summarized at Table 5.3 (a) and Table 5.3 (b).

When we look at the pH values of milk were found different every month. Although three replications were closed to each other every month. Standard deviations and average values were the proof. However, when we look at the pH values of halloumi cheese were found nearly each other every month. This was about its production.

Sample (months)		pH value		Ave	SD
January	6,80	6,81	6,82	6,79	0,02
February	6,78	6,79	6,81	6,79	0,01
March	6,80	6,78	6,79	6,79	0,02
April	6,80	6,79	6,77	6,79	0,02
May	6,79	6,78	6,77	6,78	0,01
June	6,78	6,77	6,76	6,77	0,01
July	6,79	6,77	6,78	6,78	0,01
August	6,77	6,78	6,77	6,77	0,01
September	6,78	6,79	6,76	6,78	0,02
October	6,75	6,77	6,78	6,77	0,02
November	6,81	6,82	6,80	6,81	0,01
December	6,80	6,80	6,81	6,80	0,01

Table 5.3 (a): pH values of milk samples with standard deviation and average



Sample (months)		pH val	ue	Ave	SD
January	6,35	6,34	6,36	6,32	0,02
February	6,30	6,33	6,32	6,33	0,03
March	6,30	6,35	6,34	6,32	0,02
April	6,30	6,33	6,32	6,32	0,02
May	6,35	6,34	6,35	6,35	0,01
June	6,35	6,34	6,32	6,34	0,02
July	6,33	6,30	6,36	6,33	0,03
August	6,35	6,34	6,33	6,34	0,01
September	6,35	6,30	6,35	6,33	0,03
October	6,34	6,33	6,34	6,34	0,01
November	6,35	6,33	6,35	6,34	0,01
December	6,34	6,33	6,32	6,33	0,01

Figure 5.3 (a): pH values of milk samples with standard deviation and average

 Table 5.3 (b): pH values of halloumi cheese samples with standard deviation and average





#### 5.4 Fat Analysis Results of Halloumi Cheese

The results obtained halloumi cheese fat values for 12 months in 3 replications at one time were summarized at Table 5.4.

As seen that the fat values of halloumi cheeses every month three replications were closed to each other. However each month were changed. This was about seasonal difference in milk fat.

sample (months)		Fat amou	nt %	Ave	SD	
January	24,5	24,5	24,4	24,5	0,1	
February	24,0	24,5	24,4	24,3	0,3	
March	20,8	21,0	20,9	20,9	0,1	
April	21,0	20,0	21,0	20,7	0,6	
May	21,3	21,5	21,4	21,4	0,1	
June	20,3	20,2	20,3	20,3	0,1	
July	20,1	20,2	20,1	20,1	0,1	
August	21,5	21,5	21,4	21,5	0,1	
September	21,4	21,5	21,6	21,5	0,1	
October	21,0	20,7	20,8	20,8	0,2	
November	23,5	23,3	23,5	23,4	0,1	
December	24,2	24,1	24,0	24,1	0,1	

Table 5.4: Fat amounts of halloumi cheese sample standard deviation and average



Figure 5.4: Fat amounts of halloumi cheese samples with standard deviation and average

# 5.5 Dry Matter Analysis Results of Halloumi Cheese

The results obtained halloumi cheese dry matter values for 12 months in 3 replications at one time were summarized at Table 5.5.

As seen that the dry matter values of halloumi cheeses every month three replications were closed to each other. However each month were changed. This was about seasonal difference in milk dry matter.

Sample (Months)		Dry Matter Value		Ave	SD	
January	53,45	53,50	53,50	53,48	0,03	
February	51,24	52,30	51,55	51,70	0,55	
March	49,35	49,55	49,48	49,46	0,10	
April	50,10	50,20	50,13	50,14	0,05	
May	50,23	50,13	50,24	50,20	0,06	
June	50,38	50,30	50,34	50,34	0,04	
July	49,82	49,90	49,88	49,87	0,04	
August	48,82	48,78	48,80	48,80	0,02	
September	49,88	49,90	49,92	49,90	0,02	
October	49,00	48,78	48,92	48,90	0,11	
November	52,00	52,25	52,15	52,13	0,13	
December	54,00	54,20	54,10	54,10	0,10	

**Table 5.5:** Dry matter values of halloumi cheese sample standard deviation and average



Figure 5.5: Dry matter values of halloumi cheese sample standard deviation and average

# 5.6 Sensory Analysis Results of Halloumi Cheese

The results obtained halloumi cheese sensory values which were mouth feeling, colour, odor and taste and general appearance for 12 months at one time were summarized at figure 5.6 and table 5.6.

sample	overall sensory
January	4,7
February	4,5
March	4,4
April	4,5
May	4,3
June	4,3
July	4,3
August	4,5
September	4,4
October	4,4
November	4,7
December	4,8

Table 5.6: Sensory attributes of halloumi cheese



Figure 5.6: Monthly histogram of each sensory attributes of halloumi cheese

#### **CHAPTER 6**

#### CONCLUSION

In this study, changes in fat, dry matter, pH and sensory attributes (in halloumi cheese only) of these products were analysed during the production period of one year to investigate the seasonal changes in milk and halloumi cheese. For one year, the changes in atmosphere which were temperature and moisture were observed. Monthly weather temperature and humidity forecast data were also collected. In addition to this it was found that the milk and halloumi cheeses were affected from seasonal changes in composition. In winter when total dry matter and fat content were high, sensory quality of halloumi cheeses had higher values. pH value of halloumi cheese were stable during the production period. As noticed the pH value of halloumi cheese were stable in all months, this was about halloumi cheeses production. The fat amounts max value was 24.5 % in winter and min was 20.1 % in summer. In this manner overall sensory was changed in arrange of 4.25-4.75. (Table 6.1)

Sample/	Dry	Fat %	Fat%	pН	Overall sensory scores
Months	matter	(WD)	(ab)	-	•
January	53,5	24,5	45,7	6,3	4,65
February	51,7	24,3	47,0	6,3	4,50
March	49,5	20,9	42,2	6,3	4,40
April	50,1	20,7	41,3	6,3	4,45
May	50,2	21,4	42,6	6,3	4,25
June	50,3	20,3	40,3	6,3	4,30
July	49,9	20,1	40,2	6,3	4,30
August	48,8	21,5	44,0	6,3	4,45
September	49,9	21,5	43,0	6,3	4,40
October	48,9	20,8	42,5	6,3	4,40
November	52,1	23,4	44,9	6,3	4,65
December	54,1	24,1	44,5	6,3	4,75

**Table 6.1:** Summary of some quality parameters related to halloumi cheese

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**ASLIHAN ESENDAGLI SEASONAL CHANGES IN QUALITY OF HALLOUMI CHEESE PRODUCED FROM COW MILK** 

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# SEASONAL CHANGES IN QUALITY OF HALLOUMI CHEESE PRODUCED FROM COW MILK

# A THESIS SUBMITTED TO THE GRADUATE SCHOOL OF APPLIED SCIENCES OF NEAR EAST UNIVERSITY

By ASLIHAN ESENDAĞLI

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# ÖZET

Bu araştırmada, sütte ve hellim peynirindeki mevsimsel değişimlerin kimyasal değişimlere etkisini gözlemlemek için bir yıl boyunca yağ, kurumadde, pH ve duyusal analizler yapıldı. Bir yıl boyunca, atmosferdeki sıcaklık ve nem değişiklikleri de gözlendi. Kış aylarında Hellim peynirinin üretildiği sütün, yağ değeri ve kuru madde değeri artmış, böylece duyusal olarak ağızda bıraktığı his değerlerinin arttığı görülmüştür. Hellim peynirin pH değerinin tüm aylarda sabit olduğu gözlenmiştir, bu durum Hellim peynirinin üretim prosesinden kaynaklanan normal bir sonuçtur.

Anahtar Kelimeler: hellim peyniri; süt; mevsimsel değişimler; kimyasal değişimler; kalite

To my parents...