

**ISTANBUL TECHNICAL UNIVERSITY ★ GRADUATE SCHOOL**

**ENCAPSULATION OF OIL-BASED CHEESE AROMA BY  
USING SPRAY-DRYING AND EFFICIENCY OF  
MICROCAPSULES IN MODEL FOODS**

**M.Sc. THESIS**

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**Department of Food Engineering**

**Food Engineering Programme**

**JUNE 2022**



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**JUNE 2022**



**İSTANBUL TEKNİK ÜNİVERSİTESİ ★ LİSANSÜSTÜ EĞİTİM ENSTİTÜSÜ**

**YAĞ BAZLI PEYNİR AROMASININ PÜSKÜRTMELİ KURUTMA  
YÖNTEMİ KULLANILARAK ENKAPSÜLE EDİLMESİ VE MODEL  
GIDALARDA ETKİNLİĞİNİN BELİRLENMESİ**



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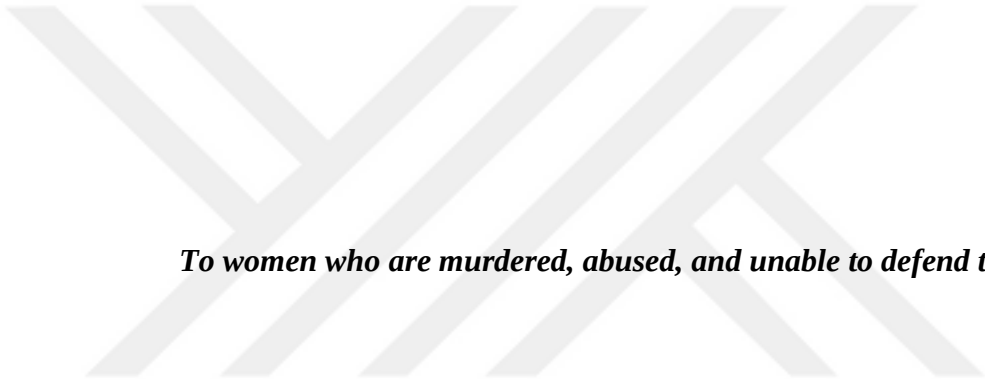
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*To women who are murdered, abused, and unable to defend their rights,*



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## **ABBREVIATIONS**

|             |                               |
|-------------|-------------------------------|
| <b>DE</b>   | : Dextrose Equivalent         |
| <b>MD</b>   | : Maltodextrin                |
| <b>SPME</b> | : Solid Phase Microextraction |
| <b>WPC</b>  | : Whey Protein Concentrate    |
| <b>WPI</b>  | : Whey Protein Isolate        |





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## **ENCAPSULATION OF OIL-BASED CHEESE AROMA BY USING SPRAY DRYING AND EFFICIENCY OF MICROCAPSULES IN MODEL FOODS**

### **SUMMARY**

Since the world has existed, food consumption has been critical for the continuation of life. Appearance, flavor, and taste play an important role in the preference of foods in nature. In modern times, these parameters have been controlled and improved. Fermentation is invented to increase the shelf-life of the food; in addition, new volatile compounds are formed during the fermentation process. Cheese is the most consuming fermented product, and different types of cheese are made by changing fermentation and process parameters. These parameters also affect the odor and taste, providing the uniqueness of cheese. Each volatile compound has a special odor and taste, therefore, containing various volatile compounds allows different cheese varieties to have their unique odor and taste. Various extraction techniques are distillation, static headspace, dynamic headspace, solid-phase microextraction (SPME), stir bar sorption extraction, and vacuum distillation applies for the extraction of the cheese volatile compounds. Depending on the volatile compounds' qualifications such as the sensitivity of heat, light, and temperature; appropriate extractions should be chosen.

The encapsulation process prevents flavors from being affected by adverse environmental conditions. Various techniques are used to encapsulate the active material. Selecting the suitable encapsulation method depends on the core material release features, wall material properties, project cost, and aim. Preparation of emulsion is the first and fundamental step of encapsulation. Wall materials that cover the core material may be consists of carbohydrates, protein, or other materials. Starch, gum, and maltodextrin carbohydrate-based derivatives to use in encapsulation; however, for getting more encapsulation efficiency carbohydrate and protein-based materials are preferred together. Both animal and plant-based derivatives are used as protein wall materials. High pressure, microtechniquesing technique, and ultrasonication are used to homogenize and provide emulsion to good dispersion. The spray drying technique is one of the most common encapsulation techniques in the food industry. The spray drying process is based on the principle of atomizing the material to be dried under high pressure and then drying it in a very short time with hot dry air.

In this experiment, commercial cheese aroma is encapsulated by the spray drying technique. Using maltodextrin and protein are used as wall material at different concentrations. 30 % of the emulsion mixture consists of solid ingredients. This solid content is prepared with 16 % aroma and 84 % mixture of maltodextrin with dextrose equivalent 11-16 and whey protein concentrate, 50 %. Protein content in the solid mixtures is 8 % and 4%. These emulsions are kept in a shaking bath at 40 C° for 20 hours at 80 rpm to improve hydration. 16 % aroma of total emulsion is added to both emulsions. After the hydration and adding aroma, the emulsion is mixed first at 450 rpm, for 1 hour; then homogenized by Ultra-turrax at 17000 rpm for 5 minutes.

Laboratory scale Buchi B-290 is used at an 8 mL/min feed rate,  $180 \pm 2$  °C inlets, and  $80 \pm 2$  °C outlet temperature. Encapsulation duration keeps 40 minutes.

Encapsulated and commercial aroma is added, a total of 1 % flavor, in cracker dough samples that include 60 % flour, 15 % oil, and other ingredients. Then the crackers are cooked in the oven at 220 °C, for 10 minutes. The aroma added cracker is oiled at % 10 without aroma. Cracker dough without aroma is prepared and cooked; however, the encapsulated aroma and liquid aroma are added in the oil process at 0.2 %.

GC & MS analysis results are compared with the results of commercial liquid flavoring as a reference. Encapsulated A and B aroma volatile compounds concentrations show similar results. Butyric acid is lost after the encapsulation process; however, 9,12-Octadecadienoic acid (Z, Z) concentration dramatically increases after the encapsulation in both encapsulated aromas. 2-Decenal and 2-Nonenal concentrations show a slight rise and these compounds are also derivatives of the oxidation. Heptanoic and Undecanal concentrations in the encapsulated aroma are higher than in the liquid flavor. Other compound concentrations are similar between A & B aromas and commercial products.

Added the encapsulated aromas and commercial aroma in cracker doughs, volatile compounds concentrations do not show dramatically difference; however, 2- Decenal concentration in three samples slightly increases with comparing the reference liquid aroma. 9,12-Octadecadienoic acid concentration is differentiated between encapsulated aroma and liquid aroma, higher concentration in the encapsulated aroma. Heptanoic acid concentration could not be maintained in doughs when using liquid flavoring and showed a decrease. After the cooking, adding the aroma to the top oil process demonstrates the different volatile compounds concentrations. Heptanoic acid, 2-Nonenal, 2-Decenal, dihydro-5-pentyl-2(3H)-Furanone, and Tetradecanal compounds concentrations are higher than reference liquid aroma and cracker that include aromas in the dough. Other compounds' concentrations are similar to the commercial aroma.

The change in the concentrations of the volatile components in the aroma may be caused by oxidation and the combination of the aroma with other components and creating different reactions. The panelists do not realize dramatically difference between using encapsulated B aroma and liquid aroma in dough with cheese odor, cheese flavor, aftertaste of cheese flavor, and general taste. Significant differences are observed between crackers that use oil including aroma and added commercial aroma in the oil process. The samples using the encapsulated B flavor in the oil are more like in terms of cheese odor and flavor, aftertaste cheese flavor, and general taste than the crackers using the reference aroma.

In line with these results, industrial cheese flavor managed to preserve most of its volatile components with its wall material consisting of maltodextrin and whey protein concentration. The encapsulated aromas are used in the dough of cracker samples, which are applied at high temperatures during cooking, and in the oil applied to them after cooking. Although some volatile compounds of crackers, such as butyric acid and 2-nonenal, are lost when high temperature is applied; a high amount of volatile components are retained. Added aroma in oil shows greater volatile compound concentrations, also added encapsulated aroma in oil is more preferred than others.

## **YAĞ BAZLI PEYNİR AROMASININ PÜSKÜRTMELİ KURUTMA YÖNTEMİ KULLANILARAK ENKAPSÜLE EDİLMESİ VE MODEL GIDALARDA ETKİNLİĞİNİN BELİRLENMESİ**

### **ÖZET**

Dünya var olduğundan beri, gıda tüketimi hayatın devamı için kritik öneme sahip olmuştur. Gıdaların tercih edilmesinde görünüm, koku ve tat önemli rol oynamaktadır. Günümüze yaklaştıkça bu parametrelerin kontrol edilebilirliği artırıldı ve geliştirildi. Fermantasyon, gıdanın raf ömrünü artırma çalışmaları yapılırken bulunmuştur ve fermantasyon işlemi sırasında yeni uçucu bileşikler oluşmaktadır. Peynir en çok tüketilen fermente ürünlerden biridir ve farklı peynirler, fermantasyon ve proses parametreleri değiştirilerek elde edilir. Bu parametreler koku ve tadı da etkileyerek peynirin kendine has tadının ve kokusunun oluşmasını sağlar. Her uçucu bileşiğin kendine has kokusu ve tadı vardır, bu nedenle çeşitli uçucu bileşikler içermesi farklı peynir çeşitlerinin kendilerine ait koku ve tadının oluşmasını sağlar.

Peynirin içerisindeki uçucu bileşikler ekstrete etmek için damıtma, static headspace, dinamik headspace, katı fazlı mikroekstraksiyon (SPME), sorpsiyon ekstraksiyonu ve vakum damıtma gibi çeşitli ekstraksiyon teknikleri uygulanır. Uygun ekstraksiyon yöntemi uçucu bileşiklerin ısı, ışık ve sıcaklık duyarlılığı gibi özelliklerine bağlı olarak tercih edilmelidir.

Enkapsülasyon işlemi, aromaların olumsuz çevre koşullarından etkilenmelerini engeller. Aktif materyali enkapsülle edebilmek için ekstrüzyon, kokristalizasyon,, püskürtmeli kurutma, dondurarak kurutma, akışkan yatakta kaplama, çeşitli enkapsülasyon teknikleri kullanılır. Uygun kapsülleme yönteminin seçilmesi, çekirdek salınım özelliklerine, duvar malzemesi özelliklerine, maliyete ve enkapsülasyon işleminin amacına bağlıdır.

Emülsiyon hazırlanması, enkapsülasyon işleminin ilk ve temel adımıdır. Aktif bileşeni kaplayan duvar malzemesi; karbonhidrat, protein veya diğer malzemelerden oluşabilir. Nişasta, sakız ve maltodekstrin karbonhidrat bazlı duvar materyalleri olmasının yanında enkapsülasyon verimini arttırmak için karbonhidratlar ve proteinlerin birlikte kullanılması tercih edilir. Protein duvar malzemesi olarak hem hayvansal, peynir altı suyu, hem de nohut proteini izolatu, soya proteini, bezelye proteini gibi bitkisel bazlı ürünler kullanılmaktadır. Yüksek basınç, mikro akışkanlaştırma tekniği ve ultrasonikasyon homojenizasyon işlemi için kullanılır ve emülsiyonun iyi dağılmasını sağlar. Püskürterek kurutma tekniği, gıda endüstrisinde en yaygın kullanılan enkapsülasyon tekniklerinden biridir. Püskürterek kurutma işlemi temel olarak kurutulacak malzemenin yüksek basınç altında atomize edilmesi ve ardından sıcak hava ile kısa sürede kurutulması prensibine dayanmaktadır.

Bu çalışmada, ticari peynir aroması püskürtmeli kurutma tekniği ile enkapsüle edilmiştir. Duvar materyali olarak farklı konsantrasyonlarda maltodekstrin ve protein kullanılmıştır. % 30 katı madde içeriğinde olan emülsiyon çözeltisi, % 16 aroma ve %

84 oranında 11-16 dekstroz eşitliğine sahip maltodekstrin ve protein karışımından oluşur.. Katı madde içerisindeki protein içeriği % 8 ve % 4 olarak belirlenmiştir. Bu emülsiyonlar, hidrasyonu arttırmak için 40°C'de, 80 rpm'de ve 20 saat sıcak çalkalama banyosunda tutulmuştur. Her iki emülsiyona toplam emülsiyonun %5'i kadar sıvı aromadan eklenmiştir. Emülsiyon gece boyunca bekletildikten ve aroma ilave edildikten sonra 450 rpm'de 1 saat karıştırılmıştır. Daha sonra T-25 Ultra-Turrax ile 17000 rpm'de, 5 dakika boyunca homojenize edilmiştir. Enkapsülasyon işlemi için laboratuvar ölçeğindeki Buchi B-290 kullanılmıştır ve emülsiyon sıcaklığı enkapsülasyon işlemi öncesi 40 °C'ye ayarlanmıştır. Püskürtmeli kurutma cihazı 8 mL/dk besleme hızında, 180 ± 2 °C giriş, 80 ± 2 °C çıkış sıcaklığında ve 100% aspirator ayarında kullanılmıştır. Kapsülleme süresi 40 dakika boyunca devam etmiştir.

Kraker numunelerinin % 60'ı un, %15'i yağ ve diğer katkı maddelerini içeren hammaddelerden oluşur ve hamur içerisine toplam aroma içeriği % 1 olacak şekilde enkapsüle aromadan ve ticari aromadan eklenir. Daha sonra krakerler 220°C fırında 10 dakika pişirilir. Son olarak krakerlerin üzerine % 10 oranında yağ püskürtülür. Hamurunda aroma kullanılan örneklerde yağlama aşamasında yağın içerisine aroma eklenmez. Hamurunda aroma kullanılmayan örneklerde yağlama aşamasından önce yağın içerisine % 0.2 oranında karşılaştırılmak istenen aromalar eklenir ve % 10 oranında krakerlerin üzerine eklenir.

Ticari sıvı aroma uçucu bileşik konsantrasyonları 1 kabul edilmiştir ve analiz sonuçları bu konsantrasyon verisi ile karşılaştırılmıştır. Enkapsülasyon işleminden sonra bütirik asit kaybolmuştur; fakat, 9,12-Oktadekadienoik asit (Z,Z) konsantrasyonu, her iki kapsüllenmiş aromada da kapsüllemeden sonra artmıştır. Oksidasyon sonucu oluşan 2-Decenal ve 2-Nonenal konsantrasyonları enkapsülasyon işleminden sonra hafif bir artış gösterir. Heptanoik asit ve Undekanal'ın kapsüllenmiş aromadaki konsantrasyonu ticari sıvı aromaya göre daha yüksektir. Diğer uçucu bileşiklerin konsantrasyonları, enkapsüle A ve B aromaları ve ticari sıvı aroma için benzerdir.

Enkapsüle aromalar ve ticari sıvı aroma kraker hamurlarına eklendiğinde, uçucu bileşik konsantrasyonları önemli ölçüde farklılık göstermemiştir; bununla birlikte, 2-Dekenal konsantrasyonu hamuruna aroma eklenmiş üç numuneye bakıldığında, referans sıvı aromaya göre düşük miktarda artış göstermiştir. 9,12 Oktadekadienoik asit konsantrasyonu, kapsüllenmiş aroma ve sıvı aroma arasında farklılık gösterir. Kapsüllenmiş aroma eklenen hamurda daha yüksek miktarda bulunur. Sıvı aroma kullanılan hamurlarda heptanoik asit konsantrasyonu korunamamış ve düşüş göstermiştir.

Ürünler pişirildikten sonra yağlama prosesine aroma eklenen krakerlerde farklı uçucu bileşen konsantrasyonları oluşmuştur. Hamur içerisine aroma girilerek hazırlanan örnekler, yağlama işleminde aroma kullanılan numunelere göre daha az miktarda Heptanoik asit, 2-Nonenal, 2-Decenal, dihidro-5-pentil-2(3H)-Furanon ve Tetradekanal uçucu bileşenleri içerir. Bunlar dışında diğer uçucu bileşenlerin konsantrasyonları ticari sıvı aroma konsantrasyonuna, 1, benzerdir.

Enkapsüle edilmiş aromalardaki uçucu bileşenlerin konsantrasyonlarındaki değişiklik, oksidasyon, aromanın diğer bileşenlerle birleşmesi ve farklı reaksiyonlar oluşturmamasından kaynaklanabilir. Hazırlanan numuneler eğitimli 17 panelist tarafından değerlendirilmiştir. Hamuruna ve yağına ticari aroma eklenen numunelere referans 5 puan tanımlanmıştır, panelistler enkapsüle B aroması kullanılarak

hazırlanan numuneleri peynir tadı, peynir aroması, aftertaste peynir tadı ve genel tat yönlerinden değerlendirmişlerdir. Ticari aroma ve enkapsüle B aroması kullanılan örnekler arasında fark çıkmamıştır.

Bu sonuçlar doğrultusunda endüstriyel peynir aroması, maltodekstrin ve peynir altı suyu protein konsantrasyonundan oluşan duvar malzemesi ile uçucu bileşenlerinin çoğunu korumayı başarmıştır. Kapsüllenmiş aromalar, yüksek sıcaklık uygulanan kraker numunelerinin hamurunda ve pişirme sonrası uygulanan yağda kullanılmıştır. Butirik asit ve 2-nonenal gibi bazı uçucu bileşikler yüksek sıcaklık uygulandığında kaybolmasına rağmen, çoğu uçucu bileşen konsantrasyonu korunmuştur. Yağlama prosesinde enkapsüle edilmiş aroma kullanılan numuneler daha fazla uçucu bileşen konsantrasyonuna sahip olmuşlardır. Enkapsüle edilmiş B aromasını pişme sonrası yağlama prosesinde kullanılan krakerler, en çok tercih edilen ürünler olmuştur.





## 1. INTRODUCTION

Food consumption is highly affected by the flavor which is the main characteristic of food. The aroma which is the most valuable parameter, taste, and sense create the flavor. Fruit, cheese, coffee, wine, and meat are considerably impacted by the aroma (Song & Liu, et. al., 2018).

Cheese is one of the most preferred products in fermented milk derivatives. Also, cheese powder and aroma are predominant ingredients in the savory industry. Cheese is produced in five basic following steps: selection of milk, preparing cheese milk by adding a starter culture, coagulation, and forming of curd and cheese. Generally, all cheese has these basic procedures, however, milk composition difference, starter culture varieties, adding more processes, process parameter differences, maturation existence, and period affect the cheese texture and especially flavor (Fox et al., 2017). All cheese has diacetyl compound, the most significant ketones derived from citrate by the action of lactic acid bacteria, and gives buttery flavor (Urbach, 1993). Cheddar cheese has salty, sweet, bitter, cooked, sulfide, nutty, fruity, animal, mushroom, and caramel descriptive tastes (Singh, Drake & Cadwallader, 2003). Methyl butanal and 2-methyl propanal have a nutty flavor and Furaneol<sup>TM</sup>, homofuraneol, and methional give the meat/bouillon aroma to cheddar cheese (Avşar et. al, 2004). Rockford cheese has a variety of volatile compounds such as ethyl butanoate, ethyl hexanoate, ethyl heptanoate, ethyl decanoate, 2,3 dimethyl pyrazine, 2,6-dimethyl pyrazine, trimethyl pyrazine, furfural and 2-furan methanol that make up the unique Rockford cheese taste (Yüccer, Operator & Mendes, 2009). Cheese, fruity, sweet, fresh, buttery, fatty, and waxy odor are included in Mozzarella cheese. Cheese flavor comes from 2-heptanone; acetic acid gives the vinegar flavor and fruity odor obtained from benzaldehyde (Cincotta et al., 2021).

A variety of distinctive analytical extraction measurements have been experimented with for cheese aroma (Le Quéré, 2004). Although, Thomsen and friends mentioned that one extraction technique does not detect aroma compounds that give sensory perception sufficiently. Distillation, static headspace, dynamic headspace, solid-phase

microextraction (SPME), stir bar sorption extraction, and vacuum distillation are generally known extraction techniques (2014). The Solvent-Assisted Flavour Evaporation method is more effective when used with the dynamic headspace method for heavy molecule extraction rather than the most volatile compounds. The solid-phase microextraction method is another widely used extraction method for volatile cheese compounds due to its rapidness, the necessity for a small sample size, and similar results; if used optimally (Sadecka et. al, 2014). Highly volatile compounds in Gouda cheese are more effectively detected than less volatile compounds by using the static headspace technique due to its insensitivity to recovery. High vacuum distillation methods are generally used with static head-space techniques (Qian & Reinecciu, 2003). In Grana Padano cheese volatile compounds are fractionated into neutral and acid fractions by using the liquid-liquid extraction vacuum distillation method (Moio & Addeo, 1998 ).

In the 1930s first microencapsulation technology is introduced by using the coacervation technique. Today's, encapsulation technique is used in a wide variety of industries such as textile, food, pharmaceutical, cosmetic, and agrochemical. Encapsulation provides the preservation, stabilization, and release control of the properties and characteristics of the active material (Carvalho et al., 2016). Various active materials are entrapped by using different encapsulation techniques. Simplicity, reproducibility, quickness, effectiveness, and easy adaptation on a large scale should be parameters of microencapsulation. Also, active and wall material physicochemical properties, release properties of active material, project's aim, and cost are the parameters of selecting the encapsulation technique (Krishan, Bhosale & Singhal, 2005).

According to conventionally stored food, encapsulation of food has several beneficial effects such as reducing bacterial growth, providing less volatility of flavor, controlling the flavor release, and improving the safety. Using a variety of technologies for encapsulation permits the regulation of the encapsulated powder size and structure. Wide varieties of wall materials are used depending on the core and coating material characteristics, releasing properties, cost, etc. Starches, maltodextrins, corn syrup solids, and acacia gums are widespread usage as a carbohydrate-based wall materials in the food industry due to their diversity and low cost (Madane et al., 2006). Having

a low emulsifying capacity and low volatile retention are negative properties of carbohydrate-based wall material (Boetral et al., 2015). Generally, carbohydrate-based wall materials are used with protein-based coating material because of increasing encapsulation efficiency and functionality. There are reasons for the use of protein as a wall material, such as its formation of both water-soluble and insoluble groups, high molecular weight, emulsification properties, and rapid solubility. Gelatin, whey protein isolate, and whey protein concentrate are the primary protein alternatives to encapsulation (Açu& Kına, 2004). Spray drying, spray chilling or cooling, extrusion coating, fluidized bed coating, liposome entrapment coacervation, and centrifugal extrusion are the encapsulation techniques that produce microcapsules. The spray drying technique is the common flavor encapsulation technique that converts liquid particles to dry particles by using atomization. (Soottitantawat et al., 2005).

### **1.1 Purpose of Thesis**

This study aims to encapsulate commercial cheese flavor by spray drying method using maltodextrin with dextrose equivalent of 11:16, and whey protein concentrate (50 %) as wall material. To evaluate the effectiveness, GC&MS profiles of aroma before and after encapsulation are examined. To investigate the heat resistance of encapsulated aromas, commercial liquid sample and encapsulated aroma are applied to cracker samples, and volatile component analysis is performed with the help of GC&MS. Crackers with encapsulated flavor and commercial flavor are evaluated sensorially in terms of cheese taste, smell, aftertaste and general taste.

### **1.2 Literature Review**

#### **1.2.1 Flavor**

The elementary requirement of humankind is food even if it is consumed only for nutritional purposes. When foods are consumed for pleasure other than getting the necessary nutritional values, their appearance and smell are the parameters that complicate the selection of foods. Physical and chemical characteristics make it alluring or disgusting. Texture and non-volatile components are important places for the acceptance of food during chewing (Lölinger, 2000). Mankind, who learned to increase the strength of foods and improve their taste, later increased the shelf life,

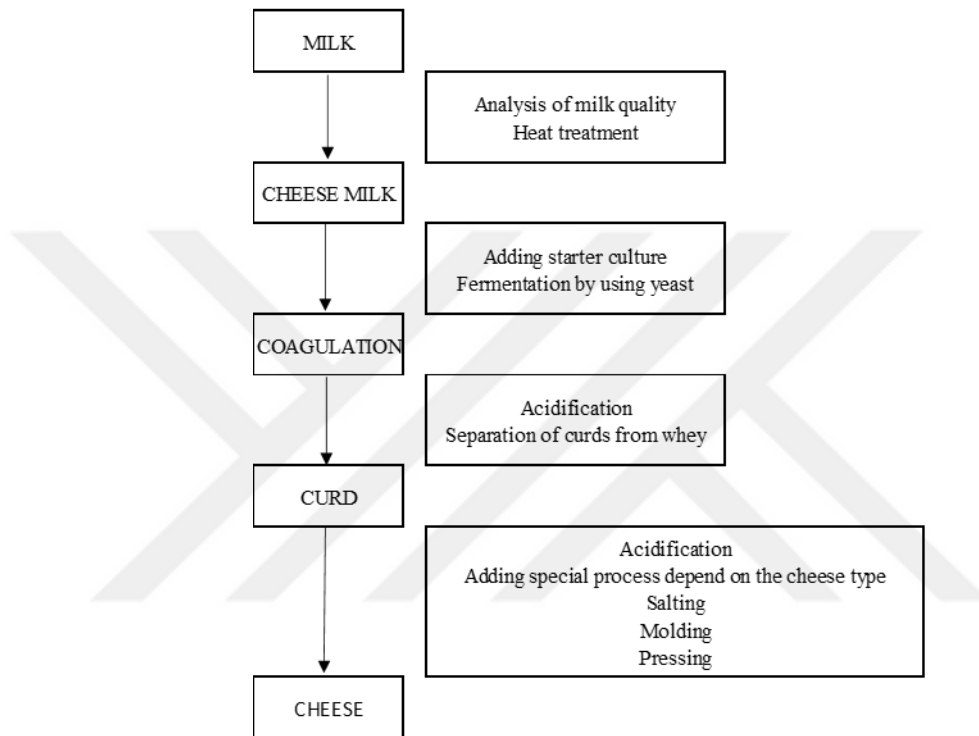
variety, and taste of foods by using food additives such as salt, spices, incense, and vinegar (Balcı Torun, 2019).

Eating food is also an entertaining social event on account of sharing food and chatting with other people (Lörliger, 2000). In the antique period the reform of sense, and odorous played a substantial role in thought, literature, ceremony, and daily life. Appearance gives fewer clues about the state of the food than smell. Incense, perfume, and nectars are used for valuable ceremonies. Aromatic plants that had scent was used for medicinal usage. (Tyler, 1996). Food flavor also is a critical compound for understanding food science even if includes a small number of volatile compounds among all metabolites in food. Research by Callingacion et al. shows that although rice contains 92 % starch and 7 % protein, tremendous variation in consumer choice and market value is associated with 1% of volatile compounds (2015). Specific organoleptic properties affect the final consumer approval. Innumerable compounds and molecules such as acids, volatile organic compounds, amines, ketones, free amino acids, phenols, alcohols, aldehydes, lactones, sulfur compounds, etc. strongly affect the organoleptic characteristics ( Tilocco et al., 2019; Fox et al., 2004)

### **1.2.2 Cheese-making and cheese flavor**

Milk-derived products are one the most consumer products in the world, especially cheese. Cheese production is based on the coagulation of milk by one of the methods that are enzymatic, acid, or acid+light, and then processing with methods that vary according to the cheese type. (Necla Aran, Food Biotechnology,2012). Cheeses have dynamic activity owing to enzymes and microorganisms so their manufacturing process has to be well organized and synchronized to obtain desirable aromas and flavors (Fox, 1993). The aroma a significant sensory feature is one of the first stimuli that affect consumption (Niimi et al., 2015). Curioni and Bosset state that a variety of functional groups and volatile compounds are included in cheese and descriptions of their functions have become objects of the extensive vast of research (2002). Cheese making generally consists of five steps (Figure 2.1). Firstly, milk is received from the farm, and then pesticides, other quality, and microbiological analyses are done. Also, pasteurization is applied in some cheese procedures; however, raw milk is also used in different types of cheese. Starter cultures are added to the milk and then to getting start the fermentation process, yeast is added. Coagulation starts thanks to acidification and

whey is separated from the curd to provide the coagulase. The salting process is important not only in increasing the development of the taste but also in protecting cheese. Giving a shape of cheese is significant for the packaging process and removing the whey. The maturation process is done in some types of cheese, as well, the duration time of the ripening depends on the desired texture and flavor of the cheese (Özdemir & Altiner, 2009).



**Figure 1.1 :** Making of cheese procedure.

#### 1.2.2.1 Cheddar cheese and flavor

Unlike other cheese productions, the most important stage of cheddar cheese is the cheddaring process. During the cheddaring application, the acidity level rises by about 0.1 % every 20 minutes and the curd blocks gain flexible and smooth structure. The curd is dried sufficiently and the acidity rises to 0.70-0.85 %. When the acidity reaches this specified value, the curd mass acquires a fibrous structure reminiscent of chicken breast meat and has a grindable feature (Üçüncü, 2008). Avşar and friend's research point out that the presence of valine, leucine, and isoleucine amino acids content provide producing Strecker aldehydes, 2/3- methylbutanal, and 2- methyl- propanal that give nutty flavor compound in Cheddar (2004). Low-fat Cheddar cheeses have a similar meat/bouillon aroma due to the high amounts of Furaneol™, homofuraneol,

and methional in the product (Milo & Reineccius, 1997). Various types of volatile compounds that give different tastes and odors are founded in cheddar cheese. Table 1.1 displays the descriptive taste and tasting sample of cheddar cheese.

**Table 1.1 : Cheddar cheese descriptive flavor.**

| <i>Descriptive taste</i> | <i>Tasting sample</i> |
|--------------------------|-----------------------|
| Bitter                   | Caffeine solution     |
| Salty                    | NaCl solution         |
| Sweet                    | Sucrose solution      |
| Cooked                   | UHT cream             |
| Sulfide                  | Etil disulfide        |
| Nutty                    | Oily peanut           |
| Fruity                   | Etil hegzanoat        |
| Animal/Barny             | p-cresol              |
| Mushroom                 | 1-okten-3-on          |
| Caramel                  | Furaneol              |

#### **1.2.2.2 Ezine cheese**

Ezine cheese is one of the brined geographically marked cheeses and is made from 40 % goat, 45 % sheep, and 15 % cow milk. Milk that is used for ezine production has to collect in Ezine / Çanakkale (Turkey). Percentages of milk required for Ezine cheese may vary according to the season, but at least 85% of the milk content must consist of goat and sheep milk. Ezine cheese production is similar to that mentioned in Figure 1.1, except bringing step. After the curd is molded, this molded cheese is added to 14-16 % brine solution (Hayaloğlu, Özer & Fox, 2008). Aroma-active components mostly aldehydes, ketones, esters, and acids are determined by gas chromatography olfactometry. Table 1.2 shows the volatile compounds and sensory description of Ezine Cheese. Some flavorings with high flavor intensity are acetaldehyde (green apple), diacetyl (butter), hexanal (cut grass), ethyl butanoate (sugar gum), dimethyl sulfide (boiled corn), (Z)-4-heptenal (oxidized oil), 1-octen-3-one (cork), acetic acid (vinegar) and butyric acid (rancid). The terms cooked, whey, creamy and fermented are also determined as characteristic sensory descriptors. (Yüceer & Operator & Mendes , 2009).

#### **1.2.2.3 Parmigiano- reggiano cheese and flavor**

Parmigiano- Reggiano is one of the brined cheese that is made from a mix of semi-creamed evening milk and morning milk, ripened for at least 1 year (Mustafa,2008).

One of the Italian cheeses Parmigiano Reggiano has a 12-24 months maturation period; sensory properties are described as 'fruity', 'sweet', and 'nut-like/roasted'. The chemicals that cause the mentioned sensory properties are as follows: butanoic, hexanoic, octanoic, and decanoic acids have cheese and lipolysis aroma; esters of ethyl butanoate, ethyl hexanoate, ethyl heptanoate, and ethyl decanoate cause fruity aroma and some pyrazines consisting of 2,3 dimethyl pyrazine, 2,6-dimethyl pyrazine, trimethyl pyrazine, furfural, and 2-furan methanol have nutty/roasted aroma (Qian & Reineccius, 2002).

**Table 1.2 :** Volatile compounds of Ezine cheese and tasting sample (Yüceer & Operator & Mendeş, 2009).

| <i><b>Volatile Compounds</b></i> | <i><b>Tasting Sample</b></i> |
|----------------------------------|------------------------------|
| Methanethiol                     | Sulphur / Cabbage            |
| Acetaldehyde                     | Green Apple                  |
| Dimethyl Sulfide                 | Sulfide / Boiled Corn        |
| 2-Methylbutanal                  | Sweet / Fruity               |
| Diacetyl                         | Butter / Cream               |
| Ethyl Butyrate                   | Fruity                       |
| Hekxanal                         | Grass                        |
| 3-Methylthiopen                  | Plastic                      |
| Heptenal                         | Leaf                         |
| Ethyl Pentanoate                 | Ester / Fruity               |
| (Z)-4, Heptenal                  | Rancid                       |
| Ethyl Hexanoate                  | Fruit / Pineapple            |
| 2-Acetyl 1-Pyrroline             | Popcorn                      |
| 2-Heptanol                       | Leaf /Metallic               |
| 2-Nonanon                        | Greasy / Dirty               |
| Methional                        | Boiled Potato                |
| Nonanal                          | Soapy / Strawy               |
| 2-Nonenal                        | Strawy                       |
| Acetic Acid                      | Vinegar / Sour               |
| Butyric Acid                     | Rancid / Cheesy              |
| (E,E) 2,4-Nonadenal              | Rancid Oil                   |
| Maltol                           | Burnt Sugar                  |
| P-Cresol                         | Barny                        |

#### **1.2.2.4 Mozzarella cheese and flavor**

Real mozzarella cheese is made from buffalo milk, yet buffalo and cow milk mix or only cow milk mozzarella cheese is done due to buffalo milk accessibility and price today. Buffalo milk has several beneficial effects to making cheese production like quick coagulation, slow acidification, late-ripening, and high yield. Mozzarella is

made up of raw milk, and brined and its production steps include boiling and kneading steps unlike general cheese production (Mustafa, 2008). Mozzarella cheese has various volatile compounds such as 2-heptanone, acetoin, benzaldehyde, ethanol, and 3-methyl-1-butanol. Other compounds are also displayed in Table 1.3. Also, the volatile compounds odor description is shown in Table 2.3 and mozzarella cheese has a unique taste thanks to all volatile compounds and odors. Volatile compounds amount significantly rise during the shelf-life by a reason of amino acids and fatty acids (Cincotta et al., 2021).

**Table 1.3 :** Mozzarella cheese flavor compounds and odor description (Cincotta et al., 2021).

| Compounds               | Odor Description                         |
|-------------------------|------------------------------------------|
| 2-Heptanone             | Cheese, fruity, ketonic, green banana    |
| Acetoin                 | Sweet, buttery, creamy, dairy, milky     |
| Benzaldehyde            | Almond, fruity, nutty                    |
| Ethanol                 | Pleasant, weak, ethereal, vinous         |
| 3-Methyl-1-Butanol      | Banana, alcohol, fruity                  |
| 2-Heptanol              | Fresh, lemon, grass, herbal              |
| 2,3-Butanediol          | Fruity, creamy, buttery                  |
| Phenylethyl alcohol     | Sweet, floral, rose-like                 |
| Acidic acid             | Vinegar, sharp, pungent                  |
| 3-Methylbutanoic acid   | Pungent, rancid, stinky, ripe fatty acid |
| Octanoic acid           | Waxy, musty, rancid, unpleasant, fatty   |
| $\delta$ -Octalactone   | Sweet, coconut, creamy                   |
| $\delta$ -Decalactone   | Sweet, creamy, coconut, milky            |
| $\gamma$ -Dodecalactone | Fatty, peach, sweet, metallic, fruity    |

#### 1.2.2.5 Emmantel cheese and flavor

Emmantel cheese has pores and is made from raw milk. The essential difference production step is propionic acid fermentation which provides a unique emmantel flavor to occur. Carbon dioxide is also released, which allows the pores to form. Its ripening period is at least 3 months. (Üçüncü, 2008). Preininger and Grosch's experiments' explained that Emmanuel cheese's 'sweety' flavor comes from metiyonal, FuraneolTM ve homofuraneol components (1994).

#### 1.2.2.6 Gouda cheese and flavor

Gouda cheese that contains small and round pores has a similar manufacturing cheese procedure mentioned above (Figure 2.1). An essential different production step is washing the coagulation to decrease acidity and lactose amount (Üçüncü, 2008).

Ninety aroma-active compounds are detected in gouda cheese by using solid-phase microextraction/gas chromatography–olfactometry. Diacetyl, 2- and 3-methylbutanal, 2-methyl propanol, methional, ethyl butyrate, acetic acid, butyric acid, homofuraneol,  $\delta$ -decalactone, and 2-isobutyl-3-methoxypyrazine are essential aroma in Gouda. (Jo et al., 2018). Shito and friends state that Gouda-type cheese has a high amount of acetoin which is ketone derivatives, ethanol, butanoic acid, and hexanoic acid. Younger Gouda cheeses have less organic acid concentrations, less fat and salt content, and higher moisture than aged cheeses. Aged cheese odor description is fruity, caramel, malty/nutty, and brothy flavors, yet younger cheese has milky, whey, sour aromatic, and diacetyl flavors (Jo et al., 2018).

#### **1.2.2.7 Kuflu/ Blue cheese and flavor**

Except for the general production step, mold culture is added with other starter cultures. If forgetting to add mold culture; after the blocking, powdery mold culture is sprinkled on the curd. 3-4 months are enough for the ripening step (Üçüncü, 2008). Ethanoic acid (acetic), Butanoic acid (butyric), and hexanoic acids are the main acids in Kuflu cheese. Esters cause the fruity flavors because of direct esterification of alcohols and carboxylic acids or via alcoholysis. Kuflu cheese has the highest concentration amount of ethyl hexanoate, ethyl octanoate, 3-methyl butyl acetate, and 3- methyl butyl butanoate. The abundance of ketones differentiates the characterization of cheese and methyl ketones which are the largest group of volatile compounds are founded in Kuflu cheese. 3-methyl butanol, 2-heptanol, and 2-nonanol include in great amounts in Kuflu cheese. Dimethyl trisulphide which has overripened cheese aroma is one of the most abundant compounds in the Kuflu cheese (Hayaoğlu et al., 2008).

#### **1.2.3 Cheese aroma and flavor extraction**

Various cheese aroma extraction methods are applied; however, one single extraction method is not sufficient for a wide range of aroma compounds extraction. Organic solvents such as acetonitrile, pentane, dichloromethane, or diethyl ether are generally used for solvent extraction. Solvent extraction methods implement the fat-free food so that fat extracted procedures like dialysis, low temperature- high vacuum distillation should be added as a preliminary procedure. Removing the large quantities of solvent while maintaining the volatile of flavor components is the challenge of the solvent

extraction method (Benkler and Reineccius 1980; Milo and Reineccius 1997; Suriyaphan and others 2001b; Zehentbauer and Reineccius 2002).

The dialysis method for cheese aroma extraction has a good recovery of volatile compounds; even though the process duration is very slow, the membrane surface entraps some volatile compounds, and the diffusion rate of compounds is different. A low-temperature high-vacuum distillation technique is applied for the extraction of a volatile compound obtained from unfatted Cheddar cheese by using centrifugation at 30000 g & 43 °C (Singh, Drake & Cadwallader, 2003). The steam distillation method is a widely used and beneficial technique that condenses steam distillate and immiscible organic solvent. Increasing the temperature is the main disadvantage of the steam distillation technique on account of the formation of thermal by-products flavor, and methyl ketones (Chaintreau, 2001). Solvent-assisted flavor evaporation is used for complex food matrices such as fruit juices, beer, and milk due to the properties of versatility, fast detection, and direct isolation (Engel et al., 1999). In static headspace, the food is directly sent to the GC column to detect the volatile compounds rapidly. The carrier gas is used for the dynamic headspace technique to flow sample and the sample has to be concentrated before the injected into a cryogenic trap or an adsorbent trap (Che & Reineccius, 1998). Solid-phase micro-extraction (SPME) is described new solventless isolation method in complex food matrices by using a single step. The necessity of a small amount of sample, in combination with other separation techniques and detection systems easy to, use and cost and time-efficient qualifications are the solid-phase micro-extraction advantageous (Kataoka et al., 2000).

#### **1.2.4 Aroma encapsulation and flavors**

The volatile compounds that create the flavor are very susceptible to degradation in the presence of air, light, humidity, high temperatures, and other ingredients in the food material, so extra attention has to be applied during food processing (Ma, Tan, Dai, & Zhou, 2013). During the food processing such as Maillard reactions, caramelization, oxidative and thermal degradation of lipids, degradation of sugars, proteins, ribonucleotides, pigments, and vitamins; aroma compounds occur by non-enzymatic reactions (Simon, Mumm & Hall, 2019). Undesired products like carcinogenic compounds and off-flavors occur in Maillard reactions, therefore, the food process has to be controlled to assist in the progress of manufacturing the savory

products. (Klevorn& Dean 2018). Simon and friends state that the lipid degradation process causes occur great amount of off-flavor. Decadienal, one of the most effective volatile compounds, is found in greater amounts in deep-fried foods. Sugars, amino acids, and lipids provide important volatile compounds in addition to that other compounds like Thiamine (Vitamin B1), ascorbic acid (Vitamin C) and carotenoids are also substantial for flavor formation. In the presence of thermal processing, these compounds are degradable and contribute to the composition of odor-active compounds (2019).

Encapsulation has a variety of beneficial applications like protecting or covering flavor and aroma components, improving the thermal and oxidative stability, to come through the restriction of the high volatility, controlling the flavor release, and enhancing the poor bioavailability (Saifullah et al., 2019). The microencapsulation process has two materials. One of them is core (active) material that is entrapped in the material or mixture system and it may be solid, liquid, or gas form. Coating material or wall, shell, or encapsulating agent coat is the core material. Microparticles with a diameter of 1-1000  $\mu\text{m}$  form multicomponent structures (Lam et al., 2010). Several encapsulated materials and coating materials are found in the literature and industry. Choosing wall material affects the encapsulated particles' physical and chemical qualifications. Active and wall material's physical and chemical qualifications, core material stability and release properties, and method of encapsulations should be taken into account for microencapsulation (Fernandes et al., 2014).

#### **1.2.4.1 Wall material**

The purpose of usage, encapsulation process, and characteristic properties of the core material is the main parameter of the selection of coating material (Saifullah et al., 2019). Good solubility, high emulsification properties, film-forming characteristics, drying features, supply low viscosity, and good covering to protect core material from oxidation are properties that should have include in wall materials (Gharsallaoui et al. 2007). Properties of the wall material not only affect the functional characteristics such as molecular structure, wall material film properties, stability of emulsification, and rheological characteristic but also influence the process parameter that are the size of the emulsion droplets, the water content, yield, efficiency, apparent morphology, particle size distribution, bulk density, and other physical and chemical properties.

Besides the above characteristic features, feed liquid made by wall material rheological qualification influences the stability, dispersibility, release characteristics, embedding rate, and other physical and chemical properties of microencapsulation; and gives the clue about the texture changes during processing (Lu et al., 2021). Table 1.4 shows the variety of wall materials that are used for flavor encapsulation and their properties.

Starch, maltodextrin, and corn syrup are preferred coating agents due to their diversity, low cost, and wide use in foods (Nedovic et al., 2011). On the contrary, carbohydrates have some disadvantages being wall materials: hydrophobic core materials, high porosity, and low emulsifying capacity that cause low microencapsulation yield and low glass transition temperature. Carbohydrates are mixed with protein or water-soluble gum; or modified chemically, generally. (Lu et al., 2021). Hogan and friends said that one wall material is not good enough to include all required properties of wall material so that mixture of carbohydrates and proteins are focus group to enhance the encapsulation features (2001).

**Table 1.4 :** Properties of wall material used for flavor encapsulation (Madene et al., 2006).

| <i><b>Wall material</b></i> | <i><b>Interest</b></i>       |
|-----------------------------|------------------------------|
| Maltodextrin (DE<20)        | Film-forming                 |
| Corn Syrup Solid (DE>20)    | Film-forming, reductability  |
| Modified Starch             | Very good emulsifier         |
| Gum arabic                  | Emulsifier, film-forming     |
| Modified cellulose          | Film-forming                 |
| Gelatin                     | Emulsifier, film-forming     |
| Cyclodextrin                | Encapsulant, emulsifier      |
| Lecithin                    | Emulsifier                   |
| Whey Protein                | Good emulsifier              |
| Hydrogenated Fat            | Barriers to oxygen and water |

Gum acacia (gum arabic) is one of the volatile compounds coating materials sought in encapsulation technology with its high solubility, low viscosity, and emulsification properties (Nedovic et al., 2011) Acacia Senegal produces gum arabic or also known as acacia gum that has acids such as D-glucuronic, L-rhamnose, D-galactose, and L-arabinose and protein content at 5 % approximately. Emulsification properties of gum arabic come from due to the existence of protein content. Gum arabic has low viscosity and above the 50 % usage in the solution can cause gel-like form dispersion. Slightly

hygroscopic gum arabic as a wall material protect oxidation and volatilization; however, price and availability may vary (Boetral et al.,2015).

Maltodextrins protect the encapsulated oil from oxidation, but their emulsifying capacity and emulsion stability are low (Gharsalloui et al., 2007). Maltodextrins are obtained from corn flour by using acid or enzyme hydrolysis and dextrose equivalents (DE) are shown as the measure of the degradation of starch polymer (Madene et al., 2006). Bangs and Reineccius searched that a variety of DE of maltodextrins affect the retention of twelve flavor compounds. DE 10 showed the best retention of flavor, nevertheless; above the dextrose equivalent, 10 (DE 15, DE 20, DE 25, DE 36.5) flavor retention decreased (1981). Also, orange peel oil is protected from oxidation by applied encapsulation with high DE maltodextrin (Anandaraman &Reineccius, 1986). Using maltodextrin with other wall materials ensures functional and effective microencapsulation (Koç et al., 2015). From one of the beginning research on encapsulation of anhydrous milk fat, whey protein with lactose is used as wall material. Combining lactose that is low molecular weight and whey protein isolate at a 1:1 ratio increases the encapsulation efficiency (Reineccius, 2019).

In addition to food hydrocolloids, food proteins such as sodium caseinate, whey proteins, and soy protein isolates also have been used in encapsulation technology. Containing water-soluble and insoluble groups together, interacting within and with a wide variety of substances, high molecular weights, the flexibility of molecular chains, and technological properties such as solubility are among the reasons for preference. During emulsion formation, protein molecules are rapidly adsorbed between the newly formed oil and water phase. Casein, gelatin, and gluten are the most used example of protein-based wall material (Açu & Kına, 2004). Gelatin obtained from animal connective or epidermal tissues collagen has good emulsification, film-forming, water-solubility, wide sources, and low price. Another protein derivative is whey protein subdivided into two categories whey protein concentrate (WPC) and whey protein isolate (WPI). WPI which is more proper as an emulsifier agent has more than %90 proteins unlike whey protein concentrate at 30-80 % (Keogh and O'kenedy, 1999).

#### **1.2.4.2 Emulsification techniques**

Two immiscible solvents are mixed in the emulsification process and the solution is known as emulsions. In encapsulation methods such as spray drying and freeze-drying, the emulsification technique is an introductory step. The most used techniques for emulsification are high-pressure homogenization, ultrasonication, and microfluidization (Laokuldilok et al., 2016;). High-pressure homogenization technique is an unproductive technique due to its necessity of high energy requirement; however, ultrasonication is a budget-friendly process with appropriate surfactant usage at low concentration. Although its cost-efficiency, the ultrasonication process requires high amounts of emulsion. Flexible control of emulsion droplet size and emulsification capacity are the preference reasons for the microfluidization technique (Kaltsa et al., 2013).

Supplying a high density of energy and the unproblematic procedure is the reason why high-pressure homogenizers are generally used for emulsification lately. On a big scale, the high-pressure homogenization technique is generally used. Firstly, the emulsion is made by a high-shear mixer roughly. Then under 100–2,000 bar pressure and high speed, the emulsion is homogenized in a narrow orifice (Saifullah et al., 2019; Piorkowski & McClements, 2014). The high-pressure homogenization technique principle is creating pressure difference (between 100-250 MPa), through the thin valve in a short time. Coarse emulsion droplets break down and get smaller because of high pressure. Emulsifiers are used to stabilize and prevent the agglomeration of small size droplets that have more tendency of agglomeration than coarse droplets by generating the film surrounding the surface of droplets (Siroli et al., 2018).

Consists of micro and nano-size droplets in flavor emulsion that can be made by ultrasonication. Forming a good emulsion droplet is the outcome of the excessive disruptive forces of high-intensity ultrasonic waves above the 20 kHz frequency (Anandharamakrishnan, 2014). Quartz crystal alternative responses of contraction and expansion cause the mechanical vibrations in probes. High mechanical vibration and cavitation in liquid emulsion are occurred by high-frequency sound waves. Unstable cavities cause the droplet to both reach the maximum limit and collapse in milliseconds. High temperature and high-pressure areas that account for the decrease of the droplet size appear during the collapsing of droplets (Maa & Hsu, 1999).

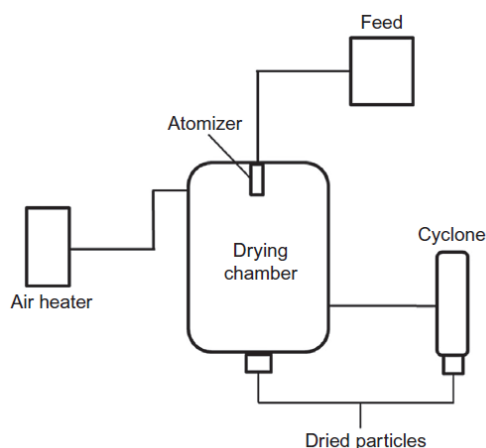
The microfluidization homogenization process is similar to the high-pressure homogenization technique except for the using channel above 20,000 psi (Ezhilarasi et al., 2013). Microfluidizer is a machine that supplies high pressure in the microfluidization process. Coarse emulsion pass through a small annular path with high pressure to break down the droplets in the continuous phase. Except for the channels where emulsion flows, high-pressure homogenization, and microfluidization has a similar design. Emulsion flows at high-pressure in two different annular cross-sectional limbs with a continuous phase. Smaller size droplets that are produced in high pressure limbs are stabilized with emulsifiers (Ezzat & Selam, 2018).

#### **1.2.4.3 Encapsulation techniques**

Encapsulation techniques are divided into two parts chemical and physical. Coacervation, molecular inclusion, and co-crystallization are included in the chemical encapsulation method. Spray drying, extrusion, freeze-drying, and vacuum drying spray cooling/chilling, fluidized bed coating are the subgroup of the physical encapsulation also known as mechanical encapsulation. Choosing the high encapsulation efficiency and loading capacity method depends on the active and wall material properties and other encapsulation parameters.

Spray drying is a well-used method of development of microparticles due to its ease to use, large-scale adaptation, give rapid results, and flexibility (Wu et al., 2014). Wall material qualification, active material properties, emulsion specification, and drying conditions are the minimum four criteria for making a better process. Airflow rate, inlet air humidity, inlet air temperature, feed rate, feed concentration, feed formulation, rheological properties, thermodynamic properties, and spray dryer specification parameters are critical for encapsulation process quality (Boetral et al., 2015). Emulsion solution preparation, emulsion dispersion that active material entrapped in wall material, homogenization, feed solution for spray drying, and dehydration of spray-dried particles are the five fundamental steps of the spray dryer process (Shahidi and Han, 1993). As shown in Figure 1.2, liquid atomization in the hot gas that converts from liquid to solid powder is the elementary principle of spray drying. (Asbahani et al., 2015). Core materials are dissolved in a coating material solution. This emulsion is put in a spray dryer chamber that has hot gas circulates for atomization, after being

sprayed into droplets. Dry droplets can be accumulated in the spray dryer chamber continuously (Carvalho, Estevinho & Santos, 2016).



**Figure 1.2 :** Equipment and machine parts of spray drying (Boetral, et al., 2015).

Accumulation of the particles on the walls of equipment is the main challenge of the spray drying process. Understanding the problem helps to decrease the deposition of particles and provides development of product quality (Keshani et al., 2015). Resultant powders with desired qualifications and cost reduction are another major challenge of spray drying (Vehring et al., 2007).

### 1.2.5 Literature review of the aroma encapsulation by using spray drying

Using flavor is essential for the food industry on account of improving the food taste and influencing the consumer. Nevertheless, food flavor compounds are very sensitive to light, air, heat, process conditions, and other environmental conditions. Spray drying is one of the most useful techniques for volatile compounds encapsulation (Potdar et al., 2021).

2-acetyl-1-pyrroline which is the major volatile compound in the rice is encapsulated with different concentrations of gum acacia: maltodextrin wall material by using spray drying. 70:30 of gum acacia: maltodextrin rate shows the best encapsulation of 2-acetyl-1-pyrroline (Apintanapong and Noomhorm, 2003). Janiszewska and Witrowa-Rajchert investigate the different wall material effects on rosemary aroma encapsulation. 25% maltodextrin (DE:10) as wall material has the highest particle dimension at  $d_{50} = 55$  nm, and small dimensions at  $d_{50} = 29$  nm are obtained from 30 % gum arabic. 20 % maltodextrin is used to increase the encapsulated flavor amount by 10 % and 30 % gum arabic was used to provide 60 % encapsulation efficiency

(2009). Encapsulated strawberry aroma storage stability and its sensory analysis in cake and chocolate have been done by Balcı-Torun and Özdemir (2021). The results of the study showed that more than 95 % loss occurred in the total flavor components in liquid form at the end of the 60-day storage period, and this loss could be reduced by half in the samples stored at 4 C° by the encapsulation process. Chocolate and cake produced with strawberry flavor microcapsules are more appreciated in terms of sensory compared to the products with the commercial aroma. The intensity of aroma is felt more especially in the foods produced when the food is produced with encapsulated flavors. This experiment also shows that flavor stability can be achieved during the processing of foods with flavor encapsulation. Mansuroğlu and her friends investigated that chickpea protein isolate as wall material is used to encapsulate the black pepper seed oil. The encapsulation efficiency of black pepper seed oil microcapsules obtained using chickpea protein isolate increased compared to the control sample without protein. A result of this study reveals the potential of maltodextrin and chickpea protein isolate to be used as carrier material in the encapsulation of sensitive products such as essential oil by spray drying method (2019). Che Man and friends investigated the effect of three different maltodextrins on durian flavor encapsulation. Durian flavor is an Asian fruit that has a unique odor. It has a strong onion-like flavor because of thiols or thioesters, and a fruity flavor that comes from ester compounds. Also, hydrogen sulfite, ethyl hydrodisulphide, and several dialkyl polysulphides, ethyl acetate, 1,1- diethoxyethane, and ethyl 2-methyl butanoate are founded in the durian fruit pulp. Maltodextrin with glucose syrup solid (DE: 12-15), fieldose PHS-15 obtained from wheat, and have 14-16 dextrose equivalent and pasilli which dextrose equivalent is 18, and supplied from potato are three wall materials to use encapsulation. The results show that three types of wall materials are appropriate for the durian flavor encapsulation (1999).

### **1.3 Hypothesis**

In this experiment, volatile compounds aim to encapsulate and increase the heat stability to high temperatures. Use aroma in the food industry that applies high temperature in the process efficiently.



## **2. MATERIAL & METHOD**

### **2.1 Material**

The commercial cheese flavor used in the study was stored under ambient conditions. The cheese flavor was supplied from the Givaudan company. Maltodextrin (Omnia, Turkey) with dextrose equivalent 11-16, and 50% whey protein concentrate (Slava, Turkey) were used as carrier material. The powder products obtained by the spray dryer were vacuum-packed and stored at +4 C°. The materials used in the analysis; are precision balance, plastic pipette, flask, beaker, glass pipette (5-10 µl), flask, funnel, magnetic stirrer, and bag.

Devices used in the analysis; Mettler Toledo ML204 precision balance (Columbus, USA), Biosan Bio Vortex V1 Mixer (Sofia, Bulgaria), T25 Ultra-Turrax homogenizer (IKA, USA), Spray dryer (Mini Spray Dryer B-290, BUCHI, Flawil, Switzerland), Dough maker (Morton, UK), Oven (Fines, Slovenia), Water bath (Mettmert, Germany).

### **2.2 Method**

#### **2.2.1 Preparation of emulsion**

Francisco's emulsion preparation method was performed with some modifications (2020). 150 grams of emulsion was prepared with a 30 % weight of the total solid content. 30 % of total solid consists of 16 % aroma and 84 % mixture of maltodextrin and protein which the maltodextrin rate changing between 76 %, and 80 % (Table 2.1). The protein + maltodextrin + deionized water emulsion were mixed at 450 rpm for 1 hour. The mixtures were kept in a shaking water bath at 40 C°, 80 rpm for 20 hours to help to hydration the material, before homogenization. After the hydration process, emulsions were stirred again at 450 rpm for 1 hour. Then, the liquid aroma was added to the emulsion at 16 %. 5 minutes of homogenization was applied at 17000 rpm. Samples were produced in triplicate.

**Table 2.1 :** Maltodextrin, protein, and aroma percentage in the solid content.

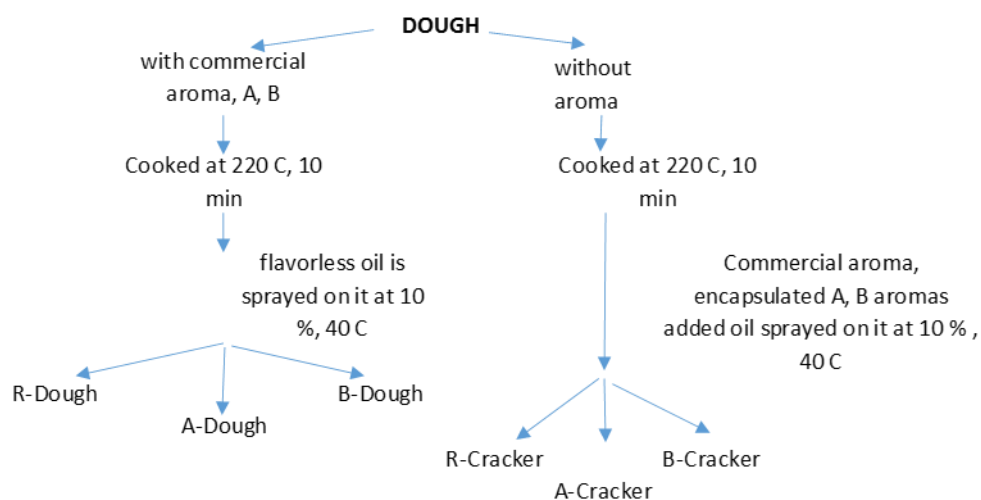
| Emulsion | Maltodextrin (%) | Protein (%) | Aroma (%) |
|----------|------------------|-------------|-----------|
| A        | 76               | 8           | 16        |
| B        | 80               | 4           | 16        |

## 2.2.2 Encapsulation process with spray drying method

Laboratory scale Buchi B-290 (Flawil, Switzerland) mini spray dryer was used in the spray drying process. The feed rate was set to be 8 mL/min. The inlet and the outlet temperatures were  $180 \pm 2$  C° and  $80 \pm 2$  C°, respectively. The aspirator was set to 100 %, nozzle cleaner 5, and the slurry temperature was set to 40 C°. Encapsulation was carried out in the spray dryer for 40 minutes. The encapsulated products in powder form were stored in vacuum packages in the refrigerator at +4 C° until the day of analysis.

## 2.2.3 Preparation of cracker sample

Cracker samples are prepared with 60 % flour and 15 % fat. Other ingredients were added to the dough for further processing of dough. The crackers were cooked at 220 C° for 10 minutes. Then the oil was applied onto the cracker at 10 %, at 40 C°. Above the process was applied in two versions of crackers. For comparing the heat resistance of encapsulated aroma, encapsulated and the commercial aroma was added into the dough at the ratio of 1 % with flavorless top oil. Another version was flavorless dough was prepared; however, encapsulated and liquid aroma are applied in oil that used in oil at 0.2 %. The aroma and oil were mixed at 500 rpm for 150 seconds to homogenize.



**Figure 2.1 :** The diagram of the preparation of cracker samples.

#### **2.2.4 Statistical analysis**

The encapsulation process was carried out in two replications and the analyzes were carried out in three parallels by spray drying method from the cheese flavor. Tukey Comparison Test was applied to the data obtained as a result of the analysis. All statistical calculations were made with the Mini Tab program (2012) and the values are given as mean  $\pm$  standard error.

### **2.3 Analysis**

#### **2.3.1 GC & MS analysis for the encapsulated aroma**

Brückner and friend's static headspace gas chromatography method is applied for the analysis of the volatile compounds of the encapsulated aroma. Optima Delta 6 fused silica capillary column, film thickness: 0.35  $\mu\text{m}$ , column length: 30 m, was used. The temperature of the constant oven was 130  $^{\circ}\text{C}$ , the column was 130  $^{\circ}\text{C}$ , the inlet temperature was 220  $^{\circ}\text{C}$  and the detector was 250  $^{\circ}\text{C}$ . Split is 15:1 and fill speed was 1000  $\mu\text{l.s}^{-1}$ , injection speed was 500  $\mu\text{l.s}^{-1}$ . The flow rate of the nitrogen was 0.5  $\text{ml.s}^{-1}$ , and the pressure was 0.765 bar (2007).

#### **2.3.2 Sensory analysis**

Sensory analyzes were carried out with 17 panelists (12 Males, 5 Females) working at ETİ Gıda Sanayi Ticaret A.Ş., aged between 23-45, who received panel training. The panelists were asked to rate samples' cheese flavor, cheese odor, aftertaste cheese flavor, and general taste of the product. Sensory analysis was performed using commercial aroma and B encapsulated flavor. R-Dough and B-Doughs were compared in themselves; aroma added oil samples, R-cracker and B-cracker are evaluated with each other. R-Dough and R-crackers' sensory parameters are set to 5 points and encapsulated aroma samples are rated between 1-10.



### 3. RESULTS & DISCUSSION

#### 3.1 Emulsion Preparation

The reference liquid cheese aroma was oil-based so to make a comparison, articles that encapsulate seed and fruit oil were used. Different dextrose equivalent maltodextrins at 4.01-7.0 and 13.0-17.0 were prepared at 10 % and 30 % concentrations for the encapsulation of rosemary essential oil. Promoting hydration, the emulsion was held in a shaking water bath at 25 C° at 90 rpm, for 18 hours. Whey protein concentration solution was used for increasing the emulsion effectivity, and two different ratios of whey protein concentration: maltodextrin at 1:3 and 3:1 was prepared. Rosemary essential oil was added three different ratios (1:40 ; 1:20 ; 1:10) (Turasan, Sahin & Sumnu, 2017) . To encapsulate cardamom essential oil, which mainly contains d-limonene, 1,8-cineole and other minor components such as terpinolene,  $\beta$ -myrcene, and pinene; 30 % whey protein isolate and 30% whey protein isolate + guar gum emulsion were prepared. Emulsions are placed at 10 C° overnight to increase hydration and show the best result for encapsulation with the 1:10 ratio of essential oil to wall material (Mehyar et al. 2014). Turasan and friend's encapsulation of rosemary essential oil study showed that whey protein: maltodextrin ratio affected both drying and encapsulation efficiencies. The ratio of the 3:1 of WP: MD showed the best drying and encapsulation efficiency regardless of maltodextrin dextrose equivalent value. Although, alteration of DE caused a change in drying rate and encapsulation efficiency; did not greatly affect the particle size distributions and surface morphologies of the encapsulated particles. Increasing the DE value (DE: 13-17) gave better drying and encapsulation efficiency than DE:4-7. 1:20 ratio of core to wall material displayed more effective encapsulation than 1:40 and 1:10 concentrations (2015). Lime and orange essential oil encapsulation, maltodextrin, and protein mixture were used as wall material. Whey protein concentrate was preferred for lime oil encapsulation yet orange essential oil encapsulation, soy protein isolate, and pea protein concentrate were combined with maltodextrin as emulsifiers. Both lime and orange essential oil's wall materials dispersion were stored for a minimum of 18 hours

to develop the hydration ( Campelo et al., 2017; Francisco et al., 2020). Another natural emulsifier, chickpea protein isolate was studied for the encapsulation of black pepper seed oil. The emulsion consists of maltodextrin and different chickpea protein isolate concentration with 8 % of black pepper seed oil (Mansuroğlu et al., 2020). In all the articles mentioned above proteins regardless of plant or milk derivatives show good emulsifier properties with maltodextrin to encapsulate the oil and oil-based core materials. In this research, maltodextrin and whey protein concentrate were mixed in emulsion preparation. 1 % and 2% protein of the total emulsion were used for the emulsion preparation, and according to GC & MS results, lower protein content showed better entrapped of the volatile cheese aroma. 16 % oil-based cheese aroma in the solid content was added to the emulsion and kept for 20 hours in a shaking water bath at 40 C° at 80 rpm.

### **3.2 GC & MS Analysis**

#### **3.2.1 Analysis of liquid cheese aroma**

GC-MS analysis was performed to determine the chemical composition of the cheese flavor before and after the encapsulation process and to examine the effect of the encapsulation process on the chemical composition of the cheese flavor. The result of the GC-MS analysis of cheese flavor was shown in Table 3.1 Butanoic acid which gave cheese and sour flavor was the most found volatile compound in the commercial cheese aroma. Cheese aroma also had 20.9 % of 2,4- Dodecadienal. Heptanoic acid and 2- Nonanone were detected in approximately the same amounts (12.6 % & 12.4 %) in liquid cheese flavor. Also, a variety of volatile compounds were found in small amounts.

The encapsulation aim was to reach the closest content to the volatile component composition mentioned above. Sigh and friends research identified the cheddar cheese aroma compounds. Different maturation times and temperature processes could affect the formation of volatile compounds. Although, Butyric acid, (E)-2-Nonenal, (E, E)-2,4-Nonadienal, 1-Octen-3-one, and Nonanal were founded in cheddar cheese (2003).

**Table 3.1 : Volatile composition of the commercial liquid aroma.**

| Compound                                        | Amount(%) | Flavor | Odor      | Odor Strength |
|-------------------------------------------------|-----------|--------|-----------|---------------|
| Butanoic acid, butyl ester                      | 31.4      | Sour   | Cheese    | High          |
| 2,4- Dodecadienal                               | 20.9      | Fatty  | Fatty     | Medium        |
| Heptanoic acid                                  | 12.6      | waxy   | Fatty     | High          |
| 2-Nonanone                                      | 12.4      | Cheese | Fruity    | Medium        |
| 2-Nonenal                                       | 10.3      | Fatty  | Fatty     | High          |
| Ethylene glycol di-n-butyrate                   | 3.4       | Sour   | Cheese    | High          |
| trans-2,4-Decadienal                            | 3.0       | Fatty  | Green     | High          |
| dihydro-5-pentyl-2(3H)-Furanone                 | 2.0       | Fruity | Creamy    | Medium        |
| 5-Hexyl-2-furaldehyde (HMF)                     | 1.0       | -      | -         | -             |
| 2-Decenal                                       | 0.8       | Fatty  | Fatty     | High          |
| Trans-2,4-Dodecadienal                          | 0.6       | Fatty  | Fatty     | Medium        |
| 9,12-Octadecadienoic acid (Z, Z)-, methyl ester | 0.6       | -      | -         | -             |
| Tridecanal                                      | 0.4       | Waxy   | Aldehydic | High          |
| Undecanal                                       | 0.3       | Waxy   | Aldehydic | High          |
| Tetradecanal                                    | 0.3       | Fatty  | Waxy      | Medium        |

### 3.2.2 GC & MS analysis of encapsulated cheese aroma

Encapsulated aroma A and B samples were not significantly different from each other (Table 3.2). The encapsulation process caused the some volatile loss. Butyric acid and 2-Nonanone (CAS) did not found in the aroma; however, 9,12-Octadecadienoic acid (Z, Z), also known as alpha-Linoleic acid, concentration was, at 6.28, higher than the liquid reference aroma. The absence of the butyric acid caused cheese and sour aroma loss in the encapsulated aroma, and having high odor strength may affect the sensory qualities. Also, heptanoic acid at 1.29 concentration; 2-Nonenal at 1.26 concentration; 2-Decenal at 1.51 concentration; Undecanal at 1.36 showed an increase assuming reference volatile concentration of 1. According to reference; trans-2,4-Decadienal, tridecanal, 2,4-Dodecadienal, 5-Hexyl-2-furaldehyde, dihydro-5-pentyl-2(3H)-Furanone, Trans-2,4-Dodecadienal, and Tetradecanal considerably undifferentiated. Heptanoic acid concentration did not fluctuate in the encapsulation process. 2-Nonenal was different between the liquid aroma and encapsulated aromas, whereas this component gave strawy flavor to mozzarella cheese. Butyric acid which gave nutty flavor was founded in a variety of cheeses such as Cheddar, Parmigiano, Gouda, and Kuflu (Avşar et. al, 2004; Qian&Reineccius, 2002; Shito et al., 2015; Hayaoğlu et al., 2008). Butyric acid components were prevented in the encapsulation process by using 3 different wall materials which were octenyl succinate starch, maltodextrin, and

octenyl succinate starch /maltodextrin; however, this article contrast experiment that preferred carbohydrate to entrapped of high molecular weight, apolar, and less volatile compounds. Containing butyric acid in encapsulated aroma depends on the relative volatility of the compound and emulsion diffusivity and interaction qualities (Mongenot, Charrier & Chalier, 2000). Cream cheese linoleic acid content significantly rose after the 4 weeks at 7 C° (Jeon et al., 2011). Changing the environmental conditions affected the linoleic acid amount in cheese, especially tended to increase.

**Table 3.2 :** The concentration of volatile components of the encapsulated aroma.

| Compound                              | Liquid Aroma | A-Encapsulated Aroma | B-Encapsulated Aroma |
|---------------------------------------|--------------|----------------------|----------------------|
| Butanoic acid, butyl ester            | 1            | 0.00                 | 0.00                 |
| Heptanoic acid (CAS) Heptoic acid     | 1            | 1.29                 | 1.29                 |
| 2-Nonanone (CAS) Methyl heptyl ketone | 1            | 0.00                 | 0.00                 |
| 2-Nonenal,                            | 1            | 1.26                 | 1.26                 |
| 2-Decenal                             | 1            | 1.51                 | 1.51                 |
| Undecanal                             | 1            | 1.36                 | 1.36                 |
| trans-2,4-Decadienal                  | 1            | 1.09                 | 1.09                 |
| Ethylene glycol di-n-butyrate         | 1            | 0.00                 | 0.00                 |
| Tridecanal                            | 1            | 1.15                 | 1.15                 |
| 2,4-Dodecadienal                      | 1            | 1.01                 | 1.01                 |
| 5-Hexyl-2-furaldehyde                 | 1            | 1.02                 | 1.02                 |
| dihydro-5-pentyl-2(3H)-Furanone       | 1            | 1.11                 | 1.11                 |
| Trans-2,4-Dodecadienal                | 1            | 1.08                 | 1.08                 |
| Tetradecanal                          | 1            | 1.18                 | 1.18                 |
| 9,12-Octadecadienoic acid (Z,Z)       | 1            | 6.28                 | 6.28                 |

Nonanal and 2-decenal were produced from oleic acid (C:18) oxidation. Hydroperoxide occurred in the oxidation process and is found in C:9 & C:10 positions in oleic acid. Hydroperoxide was not only formed at C:9-10 positions by singlet oxygen but also formed by triplet oxygen at C:8-9-10-11 in oleic acid. Nonanal and 2-decenal occurred in both oxygen types. Nonanal and 2-decenal gave off-flavor in goat cheese (Kim et al., 2003). Increasing 2-decanal concentration could be the result of the oxidation of the linoleic acid in the aroma. 2-nonenal (E, E)-2,4-nonadienal, (E, Z)-2,4-decadienal, and (E, E)-2,4-decadienal volatile compounds founded in reduced-fat Cheddar cheese with lecithin. These aldehydes were linoleic acid decomposition products in unmodified granular soy lecithin. Oxidation of the unsaturated lecithin may increase due to the high water content of the cheddar and oxidative activity of the

cheddar culture (Suriyaphan, Drake & Cadwallader, 1999). In this experiment, increased the linoleic acid content after the encapsulation process may also be elevated other volatile compounds present due to oxidation.

### **3.2.3 GC & MS analysis of encapsulated aroma**

#### **3.2.3.1 GC&MS analysis of encapsulated aroma in cracker**

Encapsulated powder aroma was applied to cracker dough to investigate the encapsulated aroma heat resistance. The powder was added to the dough so that the flavor content is 1 %. R-Dough refers to dough in which liquid sample was used. 3 sample dough only included encapsulated aroma and dough ingredients which were flour, fat, sugar, water, salt, baking powder, and flour treatment agent. These 3 dough samples' volatile compounds concentrations compared with the liquid cheese aroma. Butyric acid concentration showed in liquid cheese aroma; however, 3 dough samples did not contain any Butyric acid content. The interactions of other molecules or applied heat may cause the loss of volatile compounds. The encapsulated aroma had not to contain butyric acid so A- Dough, and B-Dough were not suitable to compare with liquid cheese aroma for butyric acid concentration. If butyric acid appeared in A-Dough and B-Dough, these volatile compounds come from dough ingredients, the interaction of molecules, or process conditions. Heptanoic acid content prevented its amount in A & B Dough, yet in R-Dough, its concentration level decreased. Decreasing the heptanoic acid happens may be a result of the heating process. The encapsulation process protected the heptanoic acid from the cooking step. 2-Nonanone (CAS) Methyl heptyl ketone was also dismissed in encapsulated powder and 3 samples of dough. 2-Decenal which occurred in the oxidation process content rose for the reason that heating promotes oxidation. 9,12-Octadecadienoic acid concentration did not increase compared with encapsulated aroma. During cooking, flavor loss was affected by various factors such as initial droplet concentration, composition, size, interfacial properties, and the composition and structure of the initial emulsions (Doi, Wang & McClements, 2019). The oil process was applied after the cooking of the dough. The oil process was not extremely high like cooking, its temperature range was between 40-50 C° so volatile compound concentrations were expected to be more retained than in heating process. Table 4.1 showed the differences in the volatile

compounds between dough that contains encapsulated aroma and end-product with oiled that encapsulated aroma was added.

2-Decenal was detected in the highest concentration in the cracker sample, which was applied encapsulated flavor in the oil process, and differs from the cracker in which the encapsulated flavor was added in dough. 2-Nonenal concentration increased when the encapsulated aroma was used in oil, however, using encapsulated aroma in cracker dough did not affect the 2-Nonenal amount showed decreasing tendency. Hydrophilic emulsion matrix and low water activity provided a suitable environment for the high flavor stability due to immobilizing the aroma flavor for volatilization and allowing the removal of oxygen (Blake, Cantergiani, & Harvey, 2003; Kindel, Krammer & Trauttsch, 2003). Spray-dried water-soluble flavor showed successful application in low moisture bakery products such as chocolates and cream fillings of baked goods (Heiderich and Reineccius, 2001). Encapsulated aroma is also applied after heating process owing to poor heat stability. Using as a dust-on aroma for savory snacks like chips and crackers and cereals and bakery coating were successful applications for encapsulated aroma; however, covering the surface with encapsulated aroma accelerated the flavor release. Having lower moisture content, volatility of flavor, and/or mass transportation in encapsulation environment than the main product was provided sustained-release. Viscosity of the flavor oil, wall material thickness, and size increased the retention. If the above conditions were applied, better flavor retention was observed (Roos, 2006).

**Table 3.3 :** The concentration of volatile components of the encapsulated aroma in the dough and oil process.

| Compounds                             | R- Dough | A-Dough | B-Dough | R-Cracker | A-Cracker | B-Cracker |
|---------------------------------------|----------|---------|---------|-----------|-----------|-----------|
| Butanoic acid, butyl ester            | 0.00     | 0.00    | 0.00    | 0.00      | 0.00      | 0.00      |
| Heptanoic acid                        | 0.46     | 1.20    | 1.10    | 1.37      | 1.31      | 1.30      |
| 2-Nonanone (CAS) Methyl heptyl ketone | 0.00     | 0.00    | 0.00    | 0.00      | 0.00      | 0.00      |
| 2-Nonenal, (E)-                       | 1.10     | 1.18    | 1.11    | 1.34      | 1.29      | 1.21      |
| 2-Decenal, (E)- (CAS) trans-2-Decenal | 1.43     | 1.66    | 1.55    | 1.90      | 1.79      | 1.86      |
| Undecanal                             | 1.19     | 1.09    | 1.03    | 1.21      | 1.05      | 1.15      |
| trans-2,4-Decadienal                  | 0.92     | 0.91    | 0.86    | 1.07      | 1.03      | 0.97      |
| Ethylene glycol di-n-butyrate         | 0.39     | 0.00    | 0.00    | 0.00      | 0.00      | 0.00      |
| Tridecanal                            | 0.88     | 1.12    | 0.94    | 1.11      | 1.27      | 1.25      |
| 2,4-Dodecadial                        | 0.98     | 1.10    | 1.02    | 1.28      | 1.23      | 1.14      |
| 5-Hexyl-2-furaldehyde                 | 0.94     | 1.05    | 0.97    | 1.16      | 1.17      | 1.08      |
| 2(3H)-Furanone,dihydro-5 pentyl-      | 1.02     | 1.21    | 1.01    | 1.46      | 1.24      | 1.22      |
| Trans-2,4-Dodecadial                  | 0.90     | 0.91    | 0.79    | 1.04      | 0.97      | 0.93      |
| Tetradecanal                          | 1.00     | 1.15    | 1.02    | 1.43      | 1.44      | 1.10      |
| 9,12-Octadecadienoic acid             | 1.21     | 1.51    | 1.42    | 0.00      | 1.37      | 1.20      |

Torun and Özdemir investigated the strawberry flavor encapsulation and applied it to chocolate and cake products. The results of the study showed that the encapsulation process reduced the aroma release by approximately 50 %, and the storage at low temperatures provided better aroma preservation. As a result of the sensory analysis, it was determined that chocolate and cakes produced with encapsulated strawberry flavor were more liked in terms of smell, taste, aroma, and general appreciation than the products produced using liquid form aroma. The results showed that the encapsulation process not only increased the storage stability but also improved the stability of the foods to which it was added during processing (2021).

### 3.2.4 Sensory analysis of crackers

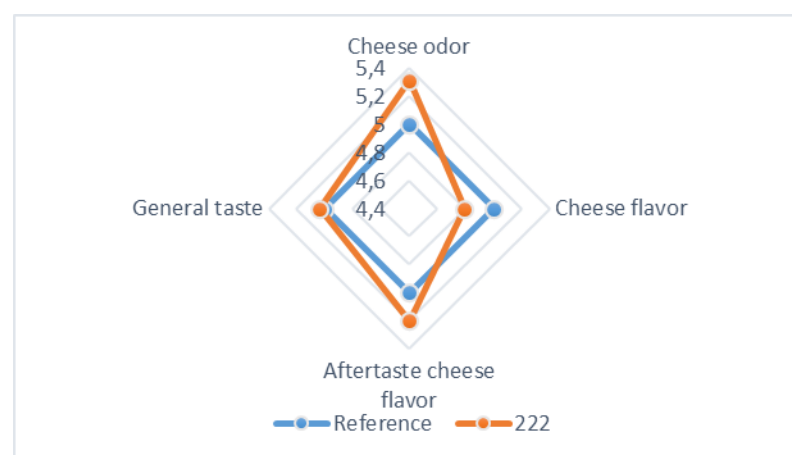
In the food industry, flavoring agents were used in bakery products, beverages, milk products, snacks and nuts, chocolates, and gums. In this experiment, encapsulated two aromas and commercial aromas were compared in cracker products within the dough and oil process.

#### 3.2.4.1 Sensory analysis of crackers that aroma used in dough

In this analysis, cracker dough was prepared with % 1 aroma. Except for aroma, other variations were the same in the two samples. After the cooking process, 10 % of the oil was applied onto the cracker yet this oil had not any aroma. At the end of the production process, cheese flavor came from the only dough. Table 3.3 showed the score of the samples and the spider diagram was given in Figure 3.1.

**Table 3.4 :** Sensory analysis of crackers that aroma used in the dough.

|                                  | <b>Cheese odor</b>       | <b>Cheese flavor</b>     | <b>Aftertaste cheese flavor</b> | <b>General taste</b>    |
|----------------------------------|--------------------------|--------------------------|---------------------------------|-------------------------|
| <b>Reference (Liquid Aroma )</b> | 5.00 <sup>a</sup>        | 5.00 <sup>a</sup>        | 5.00 <sup>a</sup>               | 5.00 <sup>a</sup>       |
| <b>222 (Encapsulated Aroma)</b>  | 5.31 ± 1.48 <sup>a</sup> | 4.79 ± 1.36 <sup>a</sup> | 5.20 ± 1.09 <sup>a</sup>        | 5.04 ± 1.1 <sup>a</sup> |



**Figure 3.1 :** Spider diagram of sensory analysis of cracker

### 3.2.4.2 Sensory analysis of cracker that aroma used in oil process

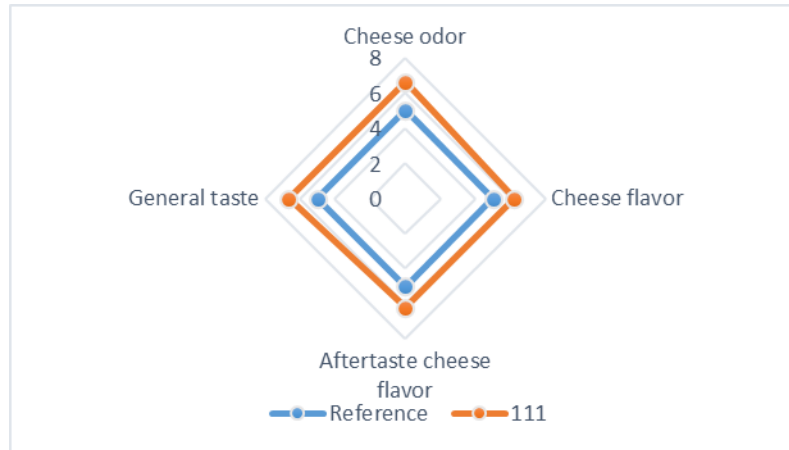
Cracker samples had not contained any aroma in their dough. Firstly, encapsulated and liquid aroma at 0.2 % dispersed in the oil. Then, the oil that included aroma was sprayed onto crackers.

**Table 3.5 :** Sensory analysis of crackers that aroma used in oil process.

|                                  | Cheese odor              | Cheese flavor            | Aftertaste cheese flavor | General taste            |
|----------------------------------|--------------------------|--------------------------|--------------------------|--------------------------|
| <b>Reference (Liquid Aroma )</b> | 5.00                     | 5.00                     | 5.00                     | 5.00                     |
| <b>111 (Encapsulated Aroma)</b>  | 6.63 ± 1.22 <sup>a</sup> | 6.18 ± 1.87 <sup>a</sup> | 6.20 ± 1.40 <sup>a</sup> | 6.64 ± 1.96 <sup>a</sup> |

The oil amount on the cracker was approximately 10 %. The sensory analysis results were shown in Table 3.4. Sensory analysis of cracker that aroma used in oil process. The panelists compared the reference and experiment sample depending on cheese odor, cheese flavor, the aftertaste of cheese notes, and general taste.

Panelists like and gave higher notes of the crackers that were oiled with encapsulated aroma. The difference between the reference and experiment sample did not show a significant difference. Figure 3.2 showed the spider diagram of the sensory analysis of the cracker that were used aroma in oil process.



**Figure 3.2 :** Spider diagram of the sensory analysis of cracker.

#### **4. CONCLUSIONS AND RECOMMENDATIONS**

Food has been an indispensable part of human history. Although it is thought that food is consumed due to the nutritional values it contains, its smell, taste, and appearance are among the biggest effects that enable the food to be consumed and preferred. Especially, the smell and appearance of the food give essential information about the consumability, attractiveness, and final condition of the food.

Fermentation technology is invented to preserve food for a long time when there is not enough technology to preserve food. In addition to the long-term preservation of foods, many foods have been discovered as a result of fermentation. Cheese is one of the most important milk-based fermentation products. Different kinds of cheese have been obtained by making changes in the milk processing process, starter cultures, and fermentation steps. Since they have different fermentation processes and production stages, different volatile component compositions create the unique taste and smell of cheese types. Various studies are carried out throughout the shelf life to preserve this smell and taste, which is the reason why it is preferred by the customer.

The encapsulation method is one of the modern methods used for the preservation of volatile compounds. The aroma, core material, which is desired to be protected, is covered with the emulsion-formed wall material. The wall material depends on the emulsion forming properties the viscosity qualifications, the film-forming characteristic, and the properties of the core material to be protected. Carbohydrates and proteins can be used alone or together as wall material. When used together, they show stronger emulsion-forming features. Starch, maltodextrin, and gum derivatives are the most commonly used carbohydrate-based wall materials in the food industry. Plant-based such as chickpea, pea, and animal-based like whey proteins are used as a wall material for encapsulation. Core material must be well dispersed in wall material to obtain better encapsulation. Various methods are applied due to emulsion properties for homogenization such as high pressure, ultrasonication, and microfluidization. A variety of encapsulation techniques is developed to use different purposes. In the food

industry, spray drying is a preferable technique due to its low cost, environmental-friendly, easy adaptation to big scale, and quick result response.

In this study, the encapsulation of industrial cheese flavor was achieved by using different ratios of maltodextrin and protein coating materials by the spray-drying method. The chemical structure of the cheese flavor was analyzed before and after the encapsulation. The concentrations of the volatile components of the reference and encapsulated flavors were compared. The most important point to be considered in the encapsulation process of aromas was the preservation of volatile component concentrations after the encapsulation process. The emulsion contained 16.6 % aroma, 80.26 % maltodextrin, and 3.34 % protein showing the best aroma entrapment.

According to the results of GC - MS analysis, the unencapsulated commercial liquid aroma had butyric acid at 31.4 %, 2,4-Dodecadialenal at 20.9 %, and Heptanoic acid at 12.6 %; 2-Nonaone at 12.4 %; 2-Nonenal at 10.3 %. Also, some of the volatile compounds found in liquid cheese aroma were below 3 % like trans-2,4-Decadienal, dihydro-5-pentyl-2(3H)-Furanone, 5-Hexyl-2-furaldehyde (HMF), 2-Decenal, Trans-2,4-Dodecadialenal, 9,12-Octadecadienoic acid (Z, Z)-, methyl ester, Tridecanal, Undecanal, and Tetradecanal. Every volatile compound had a unique flavor and odor; also, odor strength affected the cheese products' overall flavor. Encapsulated cheese aromas and volatile compounds were compared to the liquid cheese aroma. The concentration of volatile compounds in commercial products was assumed to be 1 and encapsulated aromas and cracker samples were compared to the liquid reference. Encapsulated A and B aroma results were the same. The different concentrations of maltodextrin and protein did not affect the volatile compounds' entrapment. Considering the encapsulated aromas results, butyric acid and 2-Nonanone did not preserve the encapsulation process. Butyric acid and 2-Nonenal were removed due to the encapsulation being above 150 degrees and could not be protected by the wall material. 9,12-Octadecadienoic acid dramatically increase after the encapsulation process. Heptanoic acid, 2-Nonenal, 2-Decenal, and undecanal concentrations rise due to oxidation. Oxidation causes the producing 2-Nonenal, and 2-Decenal compounds. Other volatile compounds' concentrations of encapsulated aroma were the same as the references.

The powder encapsulated aromas were added to the cracker dough. The cracker dough was prepared with 60 % dough, 15 % fat, and other ingredients. The crackers were cooked in the oven at 220 C°, for 10 minutes. Oil without aroma applied on crackers at 10 %. Liquid flavor and encapsulated flavor were added in such a way that there was 1 % flavor in the dough. After the cooking step, the volatile compound concentrations were compared with the reference liquid aroma. Butyric acid was removed from the cracker that contained liquid and powder aromas. Except for the heptanoic acid concentration, the volatile component concentrations of the three samples with flavor added to the dough were not significantly different from each other. Heptanoic acid concentration of the encapsulated aroma used cracker was greatly higher than the reference aroma used cracker. Considering the encapsulated aroma and crackers' volatile compounds concentration, values were similar to each other. According to these results, encapsulation of commercial aroma and its application in dough looked promising.

Cracker production was the same as mentioned above, except not adding any flavor to the dough mixture. Encapsulated and commercial aroma was added in such a way that 0.2 % flavor in the oil that was used on a cracker at 10 %. According to the results, crackers that used liquid aroma in the oil process did not contain any butyric acid. The powder aroma had already lost its butyric acid content during the flavor encapsulation process. Although the amount of heptanoic acid decreased when the liquid flavor was added to the dough, the amount of heptanoic acid showed similar results with samples A-cracker and B-cracker when the liquid flavor was used in the lubrication process. The Heptanoic acid concentration was similar between encapsulated aromas and cracker samples that were used in the aroma dough and oil process separately. Heptanoic acid concentration was similar between cracker samples using separately encapsulated flavor in dough and oil processing. 2- Decenal concentration of the cracker that was used aroma in oil was significantly higher than the liquid cheese aroma; however, the concentration of the 2-decenal in the six cracker samples was quite similar. The use of encapsulated aroma in the oil process did not cause any decrease in the volatile compound concentration, the compounds were preserved. Sensory analysis results show that no significant sensory difference was detected between the flavors in which the liquid and encapsulated flavor were used in the dough. In the oil process, using encapsulated aroma like more than the liquid aroma.

This study has been one of the studies that shed light on the encapsulation of aroma molecules. More work needs to be done and integrated into the food industry to develop the encapsulation of oil-based flavors. More comprehensive studies can be carried out by using wall materials in different concentrations and contents. Since temperature is one of the most important parameters in volatile component losses; product preparation with encapsulated aroma can be examined in the studies of adding oiling process and dough preparation. Applying high temperature can cause volatile compound losses; however, heptanoic acid is preserved in the dough. Added aroma in the oiling process does not show significant effect because of 40 °C process condition. Encapsulated aromas and commercial aromas' volatile compounds are preserved. The encapsulated aroma may be investigated for the dust aroma due to its low-temperature process parameter. Also, protein content rate and the source may be investigated to provide for better emulsification.

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## APPENDICES

### Appendices A:

| Groups        | N  | 1 (alpha: 0.05) |
|---------------|----|-----------------|
| Cheese Flavor | 17 | 4.1724          |
| References    | 2  | 5.0000          |
| General Taste | 17 | 5.2029          |
| Aftertaste    | 17 | 5.3106          |
| Cheese Odor   | 17 |                 |
| Sig           |    | .544            |



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### PUBLICATIONS

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